AN ANALYSIS OF SOIL AND BLOOD LEAD SAMPLES IN DELAWARE COUNTY, EAST CENTRAL, INDIANA

A THESIS

SUBMITTED TO THE GRADUATE SCHOOL

IN PARTIAL FULFILLMENT OF THE

REQUIREMENTS FOR THE DEGREE

MASTER OF SCIENCE

BY

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MUNCIE, INDIANA

May 2021

Abstract

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DEGREE: Master of Science

COLLEGE: Sciences and Humanities

DATE: May 2021

PAGES: 107

Approximately 7% of Delaware County residents have elevated blood lead levels (BLL), which is double the national average. Lead (Pb) exposure causes neuropathic effects due to its ability to replace calcium in bodily processes. Spatial analysis of BLL required utilizing blood Pb data provided by the Regenstrief Institute. Through ArcGIS Pro, spatial patterns were analyzed in Pb levels across multiple metrics. Biased sampling of soils based on legacy manufacturing and the known blood Pb data was performed to assess the presence of bio-accessible Pb in soils. Iso-concentration lines were developed based on the soil results and compared to the blood Pb data using SPSS statistics to determine any links between soil Pb and blood Pb. Results show a pattern of soil Pb contamination surrounding the historic Delco Remy battery manufacturing plant. Statistical analysis explains approximately 6.5% of factors, including but not limited to soil contact, contributing to the blood Pb issue facing Muncie citizens. This level reflects the various lead exposure pathways including water, soil, dust, vapors, familial employment in lead based businesses, or foreign imports manufactured under less stringent lead laws than those in the United States. Continued research, along with community action, is necessary to solve the continuing blood Pb problem facing Muncie.

Table of Contents

Acknowledgements

I would like to thank my advisor, Dr. Carolyn Dowling. You put forward the initial groundwork needed to acquire my dataset months before my arrival, making this thesis possible. To Dr. Jessi Haeft, thank you for encouraging curiosity and always pushing me to be a better field geologist. Dr. Klaus Neumann, thank you for reviewing my presentation abilities and offering helpful critiques, allowing improvement at every step of the way. Dr. Berland, your patience, advice, and direction proved vital in delivering the best possible maps and data for presentation. I am honored to have had the privilege to work alongside such professionals.

The Geological Sciences Department at Ball State afforded this professional a dip back into academia after having worked in the environmental industry for several years, and for that I am eternally grateful. The experience, financial support, and welcoming atmosphere of the department, including its faculty, staff, and students was one of the warmest I've experienced.

Thank you to Peter Danzl-Tauer for assisting in preparation of GIS files, maps, database analysis. Your assistance allowed significant advancement in research. A special thanks to the Fall 2018 immersive learning class for their work in sample collection.

Thank you Mom, for relentlessly pushing me to do my best throughout life and teaching me to always give 100% no matter how small the task. Thank you to my late father, who, by example, taught me to always be humble, and do my best to help and offer assistance to others whenever possible. Dr. Paul Washington, you inspired my first steps into Geology, and it means the world that you've always kept in touch and are always interested in my work. Heather, you've helped me push through the challenges of the past few years in spite of the hard times and have been a truly amazing friend and my favorite geologist.

Introduction

Lead (Pb) is a widespread contaminant found almost anywhere, from soil to older household paint, and is particularly problematic due in part to its environmental staying power (Ali et al., 2019). Pb contamination, when affecting children, can have dire health consequences, and as such, the protection of youth from significant sources of Pb contamination should be a primary societal focus (Gidlow, 2003). The percent of residents tested having an elevated Blood lead level (BLL) in Delaware County (7%) is more than double that of the national average of 2.6% (Tarrago and Brown, 2017). The purpose of this study is to characterize surface soils in and around Muncie, Indiana to discern the presence and potential concentration of bio-accessible Pb, and infer whether or not soil-lead levels are a contributing factor to the elevated BLL of local citizens. Due to the confidential nature of the blood Pb data being analyzed, all research was conducted under an approved Institutional Review Board Protocol #1181099-2.

Pb is a known neurotoxic poison, which enters the body either via ingestion or respiration, with the former being the primary route of exposure (Leggett, 1993). For children aged 5 or younger, the toxin is particularly dangerous due to their high metabolism (Leggett, 1993). When compared to adults, children's gastrointestinal systems absorb Pb in greater volumes, leading to high systemic BLL and subsequent damage to their developing brains (Lidsky and Schneider, 2003). Additional effects of Pb poisoning range from weakened bone structure and neuropathy to brain damage and death (Needleman, 2003). At more than double the national average, Delaware County's BLL problem is cause of significant concern.

This thesis will discern the presence and potential concentration of bio-accessible Pb in soils and infer whether or not soil-lead levels are a contributing factor to the elevated blood Pb concentrations of local citizens. The study will demonstrate the presence of contamination zones encompassing a former industrial site in southern Muncie. The site was home to a transmission

manufacturing facility as well as a lead-acid battery manufacturing plant (CRA, 2014). The soils beneath the former battery plant are heavily contaminated, with Pb contamination having spread off the property (CRA, 2014).

Sources of Lead Contamination

The cause of elevated BLLs is inherently difficult to examine due to the many potential sources of contamination including, but not limited to, historic vehicular emissions, lead-based paint, and industrial byproducts. Beginning in the early 1920s, Pb was utilized as an additive in gasoline to reduce engine knocking and increase power transfer in vehicles (Rosner and Markowitz, 1985). The Pb was introduced in the form of tetraethyl Pb, which when included in the fuel mix, eliminated the chance of the fuel igniting prematurely, also known as "knocking" (Rosner and Markowitz, 1985). In spite of the known adverse health effects caused by Pb, its use continued (Rosner and Markowitz, 1985). By 1973, the United States Environmental Protection Agency (EPA) began to phase out Pb from gasoline of all grades. By 1996, the EPA banned Pb for use in all on-road vehicles, while off-road uses such as aircraft and race cars are still permitted to use leaded fuel (EPA, 1996b).

Prior to its ban in 1978, Pb was utilized as an additive in paint, affording it durability and weather resistance. While no longer actively utilized, lead-based paint is still prevalent and deteriorating in older American homes numbering between 77 to 81 million as of 1990 (EPA, 1995). Following years of remediation, rehabilitation, and reconstruction the number of these structures fell by approximately 70% to a predicted 24 million ± 2.7 million units as of 2002

(Jacobs et al., 2002). The paint degrades over time with the resultant dust containing large amounts of Pb either contaminating soils or accumulating indoors (Jacobs et al., 2002).

Past industrial use of Pb includes the production of ammunition, water pipes, leaded glass, ceramic glazes, as well as in paint and gasoline (Kropschot and Doebrich, 2011). One of the largest consumers of lead was and still is lead-acid battery production, which in the early 2000s accounted for approximately 88% of United States Pb consumption (Kropschot and Doebrich, 2011). Other current industrial uses of Pb are in oxides used in glass and ceramics manufacturing, in casting processes, and for sheet lead (Kropschot and Doebrich, 2011). Additionally, Pb ammunition is still in production, though alternatives such as copper or steel based ammunition are available (Kropschot and Doebrich, 2011; Thomas, 2013).

Health Hazards

When Pb is metabolized in the human body, it bonds strongly to sulfur-hydrogen groups in proteins, which under normal circumstances would bond to calcium (Needleman, 2003). Pb acts as an inhibitor, blocking calcium from entering cells. It infiltrates mitochondria and causes swelling of the cell, preventing stimulated neurotransmission while enhancing spontaneous neurotransmission (Needleman, 2003). This replacement of calcium with Pb is the primary reason for its toxicity. BLL measurements are only useful for individuals having been exposed to a contamination source within several weeks of the test due to natural body processes removing the substance (Leggett, 1993). Chemical chelation therapy can remove Pb and other metals from an individual's blood, with the caveat that concentrations in other organs and bones can remain higher, over time releasing the stored Pb (Lowry, 2010). Primary storage of Pb occurs within the skeletal structure, with larger bones containing higher amounts of Pb contamination (Lowry, 2010). By the time Pb is detected in blood, serious neurological

damage is already done to the brain. The half-life of Pb in the brain is approximately two years, lengthening the time of neurological damage (Leggett, 1993). This being said, the only way to truly prevent lasting damage from elevated BLL is to never be exposed to Pb in the first place.

Bioavailability, Bioaccessibility, Environmental Availability, and Total Content

Bioavailability is defined by the EPA as the portion of an ingested sample or dose that is absorbed through the gastrointestinal system and is able to be utilized by the rest of the body (EPA, 2017). Bioaccessibility is an *in vitro* (located within an artificial environment) determination of the amount of a material that an organism can absorb (EPA, 2017). Environmental availability, in the context of this study, is defined as a metal's likelihood of interacting with other parts of the environmental system, with said interaction being heavily dependent on current and changing conditions (McGeer et al., 2004). A measurement of environmentally available metals denotes the entirety of metals within a sample that can become bioavailable (McGeer et al., 2004). In these terms, bioaccessibility is considered to hold the same meaning as environmental availability. This study utilized Solid Waste (SW) 846 EPA method 3050B, which outlines its ability to detect metals which may become environmentally available (EPA, 1996a). See Appendix A for the referenced EPA method write-up. Determining the extent of bio-accessible metals is important as Pb uptake in organisms occurs both by soil contact and ingestion. A further potential source for contamination includes consuming plants grown in soils with the metals present. A bioaccessibility study is able to encompass these routes of exposure.

The final term, total content, is defined by the complete amount of a constituent within a sample regardless of its capacity to interact with the environment or biota. Total Pb and bio-accessible Pb are not interchangeable phrases; however, the two terms are relatable. A study comparing the two analytical methods for Pb in Philadelphia, Pennsylvania, found a difference of only 7%

between total and bio-accessible soil-Pb content in contaminated soils (Bradham et al., 2017). The study found that total soil-lead accounted for approximately 23% of the factors contributing to a child's BLL, while bio-accessible Pb concentrations were able to predict 26% of the factors that contribute to a child's BLL (Bradham et al., 2017). While the study deemed the increase in accuracy for prediction purposes statistically significant, a 7% difference between analytical protocols for Pb content in soils at relatively low Pb concentrations can allow for qualitative comparisons in non-critical cases.

City of Muncie's Industrial History

Beginning in the late 1800s, Indiana experienced a gas boom, which brought significant economic prosperity to the region in the form of heavy industry. According to the Delaware County Government office information page, Muncie was once home to a number of heavy manufacturing industries including automotive factories, steel production industries, and glass manufacturing plants (Delaware County, 2019). The majority of larger national industries left Muncie between the 1970s and mid-2000s (Delaware County, 2019). Many of these historic facilities were located within the southern portion of the city footprint, having both major highway thoroughfares and railway spurs as a means to feed the vast material requirements of the industries. The Manual Transmission of Muncie factory and Delco Remy battery facility are among those demolished factories, leaving vacant lots in their wake. The Delco Remy battery manufacturing plant operated from 1904 until it was decommissioned in 1978 (CRA, 2011). A satellite photo displays the location of the former Delco Remy plant and Manual Transmission of Muncie factory in relation to the study area (Figure 1). Current industry includes Progress Rail, and Exide – a battery disposal facility (Delaware County, 2019). A map of current and former industry in the Muncie area is included as Figure 2.

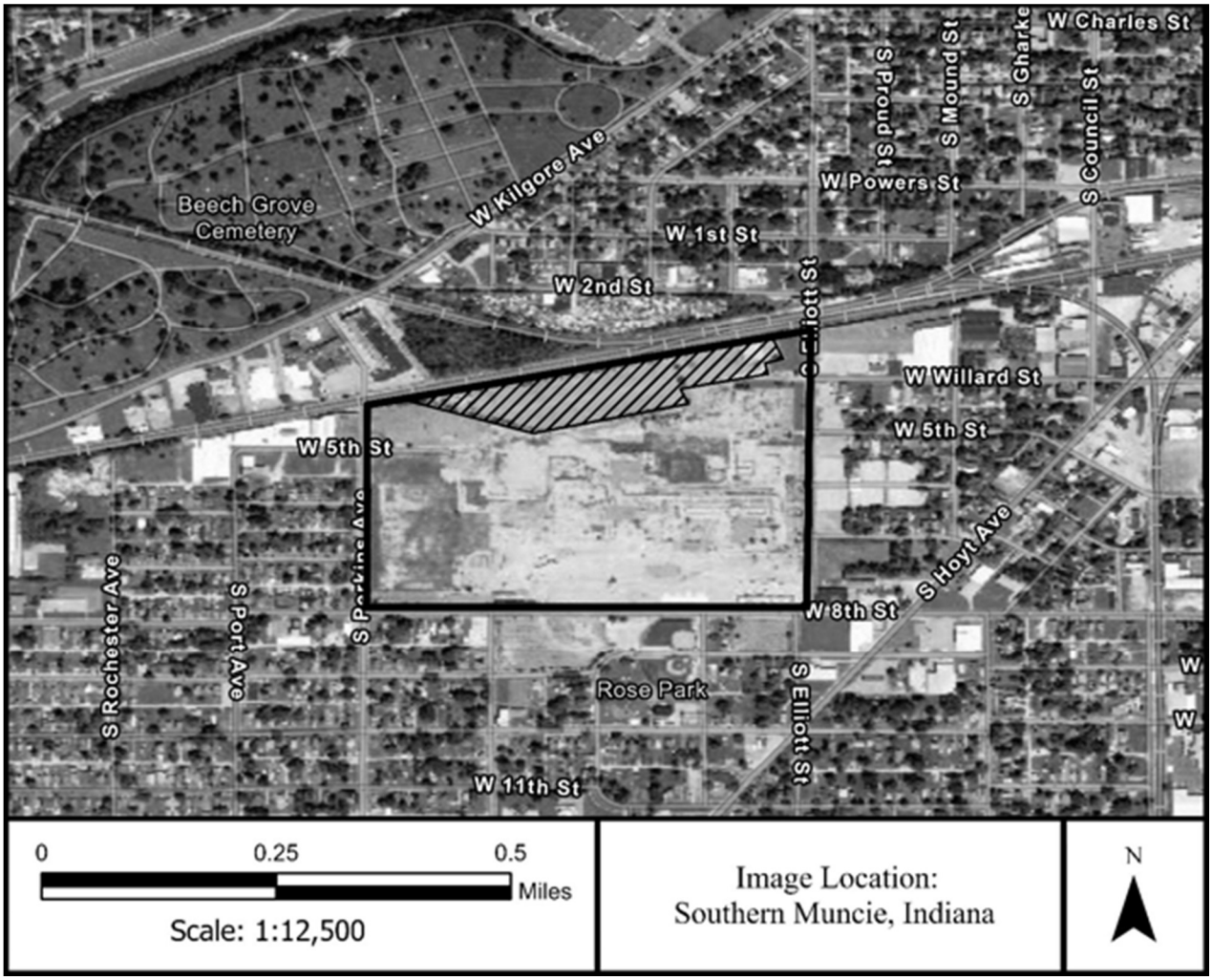

Figure 1. Satellite map of demolished former Delco Remy battery plant (hatch marks) on the former Manual Transmissions of Muncie LLC factory grounds (outlined in black).

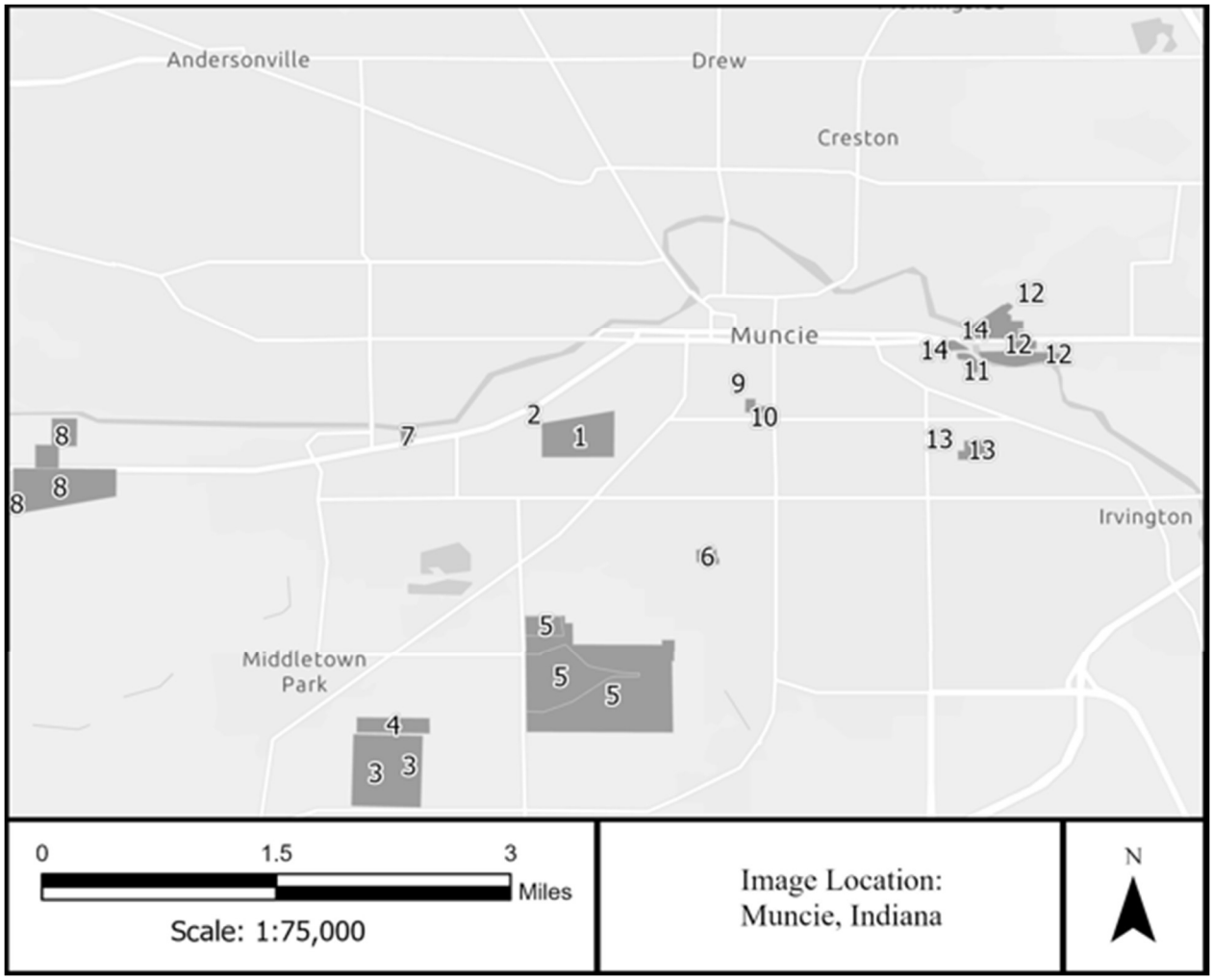

Figure 2. Shows current and former industries in the Muncie area.

1. Manual Transmissions of Muncie LLC
2. Custom Chrome and Re-Manufacturing
3. Delphi Energy & Engine
4. Exide Battery Disposal
5. Progress Rail Manufacturing Corp.
6. WITT Industries Galvanizing
7. West Killgore RCF
8. Borg Warner
9. Millennium Place III/IV
10. H&L Plating & and Grinding
11. Manufactured Gas Plant
12. Mock's Pond Landfill
13. Frank's Foundry
14. King's Forge

Previous Studies

Previous studies focusing on soil Pb in Muncie are limited in scope or measured Pb as a portion of a greater survey. A grid based sampling methodology was utilized to obtain metal concentrations across the entirety of Delaware County in a study titled Spatial Distribution of Heavy Metals in Center Township, Delaware County Surface Soils (Liberti, 1998). This study utilized sequential extraction of metals from the soils and determined that the lead and other metals contained within were not overly mobile (Liberti, 1998). Further, the findings of the study determined that significant contamination of lead exists throughout the soils of Muncie, Indiana (Liberti, 1998). While important, the study utilized a much more widely distributed sampling protocol than that used in this thesis and is not suitable for direct comparison to this study's selective higher-local point-density sampling. A more recent gardening study conducted by Obrycki (2012) differs from the current thesis in sample collection methodology and sample analysis. Sample collection was biased toward homes having gardens, and analysis of samples was only undertaken at a homeowner's request (Obrycki, 2012). Analytical methods included DTPA (a chelating agent for heavy metals) extraction for bio-available Pb and a total lead extraction using EPA method 3051a (Obrycki, 2012). Variation in sample locations occurred because gardens were located in all parts of yards including directly adjacent to older homes, potentially shedding lead-based paint chips (Obrycki, 2012).

An active Indiana Department of Environmental Management (IDEM) environmental investigation is currently being conducted on the former Manual Transmission LLC facility grounds in southern Muncie (Figure 1). The Delco Remy battery plant conducted operations including all aspects of battery manufacturing such as Pb reclamation and oxide manufacturing, assembly of battery components, case cleaning, and grinding. Slag containing

Pb was stored outside the plant on bare ground (CRA, 2011). Environmental action on the former plant grounds and surrounding residential areas found Pb in soils of public right-of-ways in 2011 (CRA, 2011). Results of this study found "total soil-lead via SW845 method 3050" at concentrations of 223 ppm upwind of the former Delco Remy facility and 2,200 ppm downwind of the property (EPA, 1996a; Webb, 2019a). It should be noted that the measurements conducted by CRA are for environmentally available Pb, rather than total Pb (EPA, 1996a). The documented SW845 is not an actively utilized code, with the proper series as follows: "SW846 EPA method 3050b." A total Pb sample was obtained from the surface of the roof of the Delco Remy facility during its decommissioning process in 1978 and was found to contain 570,000 ppm Pb (Webb, 2019a). A bio-availability study was undertaken by the Revitalizing Auto Communities Environmental Response (RACER) Trust to define a safe 'site specific closure' level for the metal contamination found (CRA, 2014). The site specific screening level for bio-available Pb for a commercial or industrial worker was listed as 1,660 ppm for direct contact to the soil by CRA and accepted by IDEM (CRA, 2014). The 1660 ppm level is a safety threshold for workers in protective gear rather than for keeping children safe. It should be noted that this bio-availability test was calculated rather than measured from site soils (CRA, 2014). In addition to the bio-availability study, off-site soil samples were taken in public right-of-ways to determine if any Pb migrated off the former factory grounds. Upwind bio-accessible soil-lead levels were reported at approximately 400 ppm, while some downwind samples northeast of the former plant were reported at over 2000 ppm (CRA, 2013). See Figure 3 for a map of estimated sampling locations and bio-accessible soil-lead levels. Aerial distribution of Pb contamination was the likely source, based on the IDEM and RACER Trust sampling methodology including wind rose data, the notable Pb concentration from the Delco Remy roof in 1978, and elevated sample concentrations downwind of the facility (CRA, 2013).

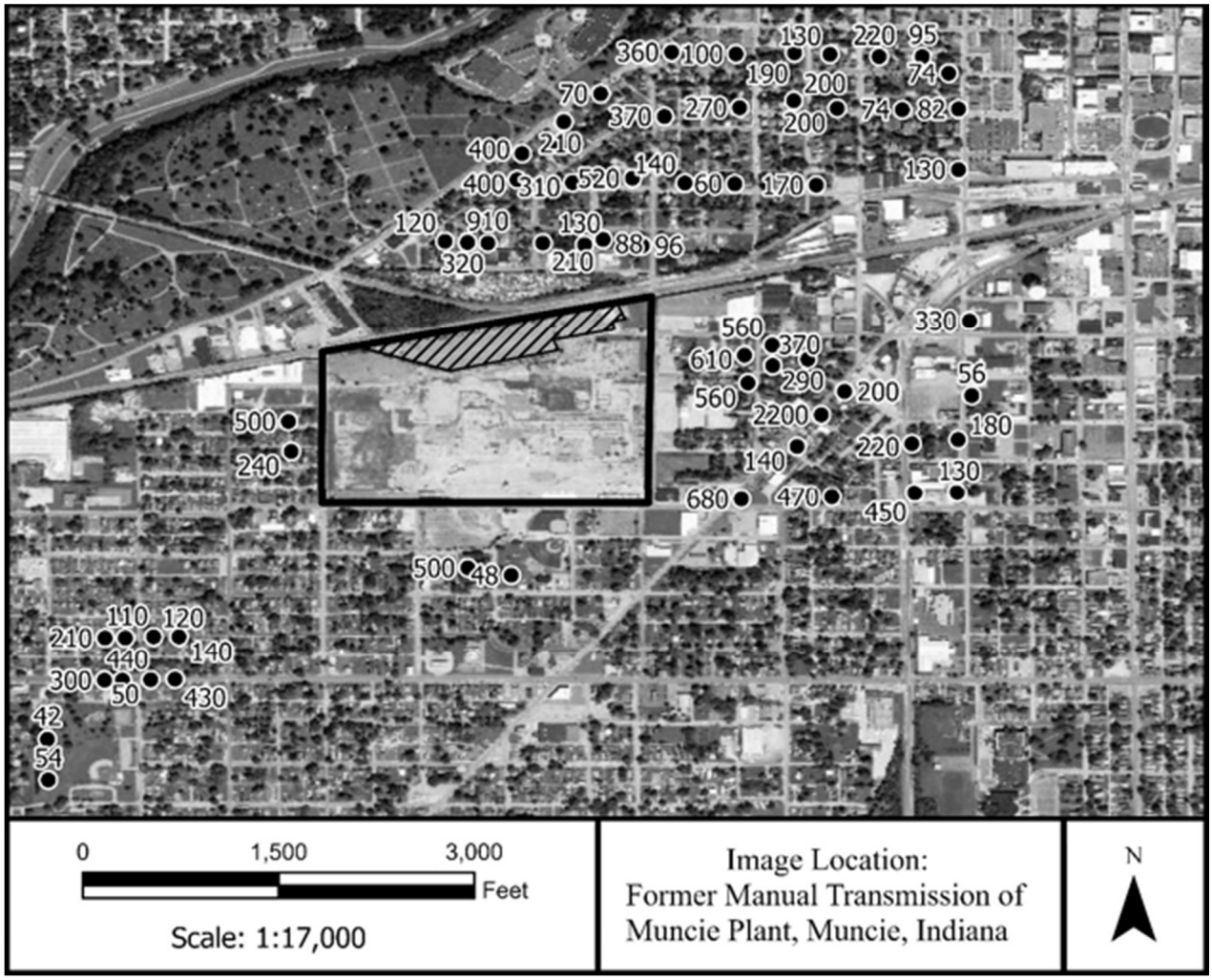

Figure 3 outlines locations sampled and analyzed by Conestoga Rovers and Associates in conjunction with a 2014 environmental site clean-up.

In September of 2014, IDEM expressed increased concern for residential Pb impacts and determined that the HUD suggested total Pb screening levels of 1200 ppm and 400 ppm for covered and bare play areas for children respectively are too high for the off-site investigation (Stilz, 2014). IDEM pushed for a blood Pb analysis to occur in conjunction with the off-site soil-lead investigation to determine the extent of the contamination (Stilz, 2014). As of

February 2020, it is unknown whether or not the BLL screening occurred based on current documentation available on the IDEM Virtual Filing Cabinet (VFC). On March 15, 2015, RACER abandoned the investigation of off-site (residential) soil-lead concerns in favor of completing on-site clean-up activities in full (RACER, 2015). However, in October 2019, IDEM determined that further investigation of soil-lead on at least 200 residential properties surrounding the former Delco Remy battery plant was necessary (Webb, 2019b). The IDEM investigation will utilize X-ray fluorescence technology to quickly analyze samples, along with lab confirmation of 10% of sample locations (Webb, 2019b). Information regarding this environmental investigation continues to become available as the investigation progresses. The history of the site and investigation is posted to IDEM's VFC (IDEM, 2020).

Methods

Database Correction and Editing

Blood Pb test results for the residents of Delaware County, IN were provided by the Regenstrief Institute in the form of an excel spreadsheet. Before any mapping could occur, the database required screening to remove incompatible test types, reporting issues, and invalid entries. All test types that indicated processing of urine and entries containing no data were removed. The urine test results consisted of only 0.5% of the total reports and were excluded for consistency. Values reported with a less than symbol were not included in mapped results. The age at test date for each individual was calculated in excel by subtracting the individual date of birth from the Pb test date and dividing the resulting number by 365 and rounding down. Several results were removed from the database due to incorrect reporting of dates of birth resulting in a negative age.

Utilizing the US Census Bureau dual-range (both sides of the street) street data and ArcGIS Pro's address locator (geocoding tool), an accurate map of BLL was generated based on the address information within the database. The initial run of the tool matched 90% of available data points, with 10% requiring fixes to the spelling of street names or zip codes.
Errors requiring the removal of points from the database include addresses being located outside of the study area, provided as an email address or post office box, or the address not matching an existing location. Following the corrections made in the first run, the geocoding tool was re- run with the amended dataset. Further corrections were required, and the third run resulted in 99.2% of data points matched, 0.73% unmatched, and 0.07% tied (duplicate points). The fourth and final geocoding run resulted in 99.34% (1360) matches, eight unmatched points requiring manual map placement, and one tie. The eight unmatched points were manually added to the map, with the tie being deleted as it matched no reasonable address location. The 'seasons' field was

populated by selecting months linked to each season: March through May as Spring (1), June through August as Summer (2), September through November as Fall (3), and December through February was classified as Winter (4).

Obfuscation of HIPPA Protected Data

To protect the individuals whose health data is contained within the database, the original point locations were obscured by a method known as 'donut masking.' Donut masking takes the coordinates of a point and shifts them away from its center in a random direction that is between a user-set minimum and maximum value, essentially creating a donut ring where a point may be placed (Zandbergen, 2014). To complete this process in ArcGIS Pro, a buffer measuring 300 meters was placed around each point, which created a polygon feature. The "create random points" tool was then utilized within each polygon to generate the new point's location with a minimum distance of 30 meters (10% of the total potential distance) from the original location. The final step in securing the new obscured points was adding the original data without address information, which was conducted by adding x, y (grid location), z (elevation), and m (tolerance) to the new table. After exporting the table to a geodatabase, the old database table was joined to the new points based on SID (identifier for each point). Lastly, all identifying address information was removed from the database.

GIS Map Preparation

A poverty database was downloaded from Census Bureau's now defunct American FactFinder for 2014 along with block group and census tract polygons from TIGER/line "topologically integrated geographic encoding and referencing datum" (Bureau, 2014, 2018). The dataset was placed into the correct block group and/or census tract following a poverty percentage calculation. A housing-age database was downloaded from American FactFinder for

2017, which utilized the same block group/census tract set as before (Bureau, 2017). This database was split into housing constructed pre and post-1980, giving a two-year buffer to the ban on lead-based paint, which occurred in 1978. The percentage of houses constructed prior to 1980 was then calculated for and assigned to each block group.

Data for industrial parks, brownfields, and underground storage tanks were downloaded from the publicly available IndianaMap (https://www.indianamap.org/) resource and added as a map layer. The Underground Storage Tanks (UST) map data were edited, and all tanks not designated as "LU" or leaking underground were removed, leaving only points of contamination.

Sample Point Selection

Utilizing a preliminary BLL map, soil collection points were selected based on the location of blood Pb test results greater than or equal to ten micrograms per deciliter. Ten micrograms per deciliter was chosen due the increased number of individuals in comparison to the ≥ 25µg/dL metric. Similarly, the ≥ 5µg/dL group was discounted due to the comparatively large number of tested individuals. The resulting group within the ≥10µg/dL series allowed for an accurate identification of potential soil-lead sources. Primary points of interest included public parks in proximity to these individuals. Additional locations close to former or current industrial zones were further utilized. A total of 25 locations were initially selected for sampling in the fall of 2018, with an additional location added in the summer of 2019. One sample location was set to act as a control, identified as Ginn Woods, in the northern part of Delaware County (Figure 4). This old-growth forest will serve as background levels for soil-lead. In total, 88 samples were collected over the 26 locations. See Appendix B for the individual site maps generated.

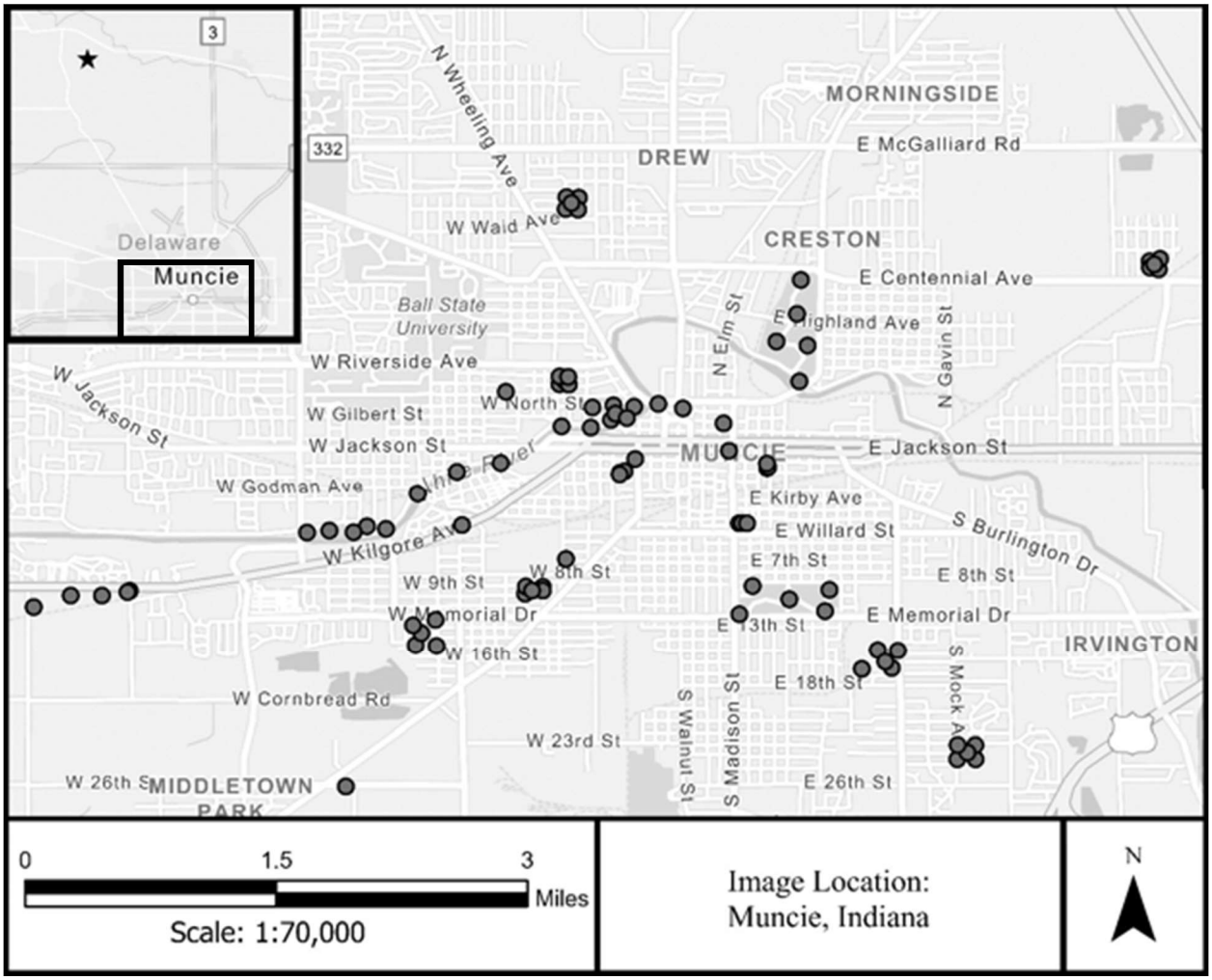

Figure 4. Map of Muncie area containing soil sample locations; the inset map shows the location where two background samples were obtained from an old-growth forest. The inset map black box outlines the sample area for primary Muncie samples.

Field Sample Collection

Sample collection was conducted using a modified Housing and Urban Development (HUD) sampling methodology. The HUD method typically investigates residential properties in various locations from the edge of a home to the rest of the yard (HUD, 2012). The original sampling procedure includes the top half-inch of soil, which is commonly collected via scraping a laboratory-clean sample vial across the area to obtain sample material (HUD, 2012). Public parks and road-side samples were collected with a hand-operated direct push soil probe to a total depth of two inches. Where possible, the layout of the five samples consisted of the four corners of a property and a central point (see Figure 5 for a diagram outlining the soil sampling methodology). Corner samples were taken ten feet from the corner of the property. Each sampling point consisted of nine sub-samples spaced approximately two feet from one another in a grid. With enough space on a property, five sample points were utilized; however, the number of collected samples was decreased when the area was limited in size. A "large" site contained five sample points. Smaller sites, those with significantly less area or when a site was irregularly shaped, were sampled at roughly equal intervals along the length of the site. Sites that were extremely small, such as a small dog park or memorial parks contained only one sample. The final determination of sample numbers occurred in the field, based on an on-site survey. During the collection process, organic material such as grass or leaves was removed prior to using the soil probe (HUD, 2012). Soil sub-samples 1-9, as per Figure 5, were placed into buckets and mixed to homogeneity before their transfer to labeled sample baggies. Two duplicate samples were submitted to the laboratory to aid in determining the test accuracy of the lab. To prevent cross-contamination of samples, a three-part decontamination system consisting of a primary liquinox wash and two secondary rinsing buckets were utilized alongside dedicated scrub brushes. Soil probes were subjected to decontamination between all sample points.

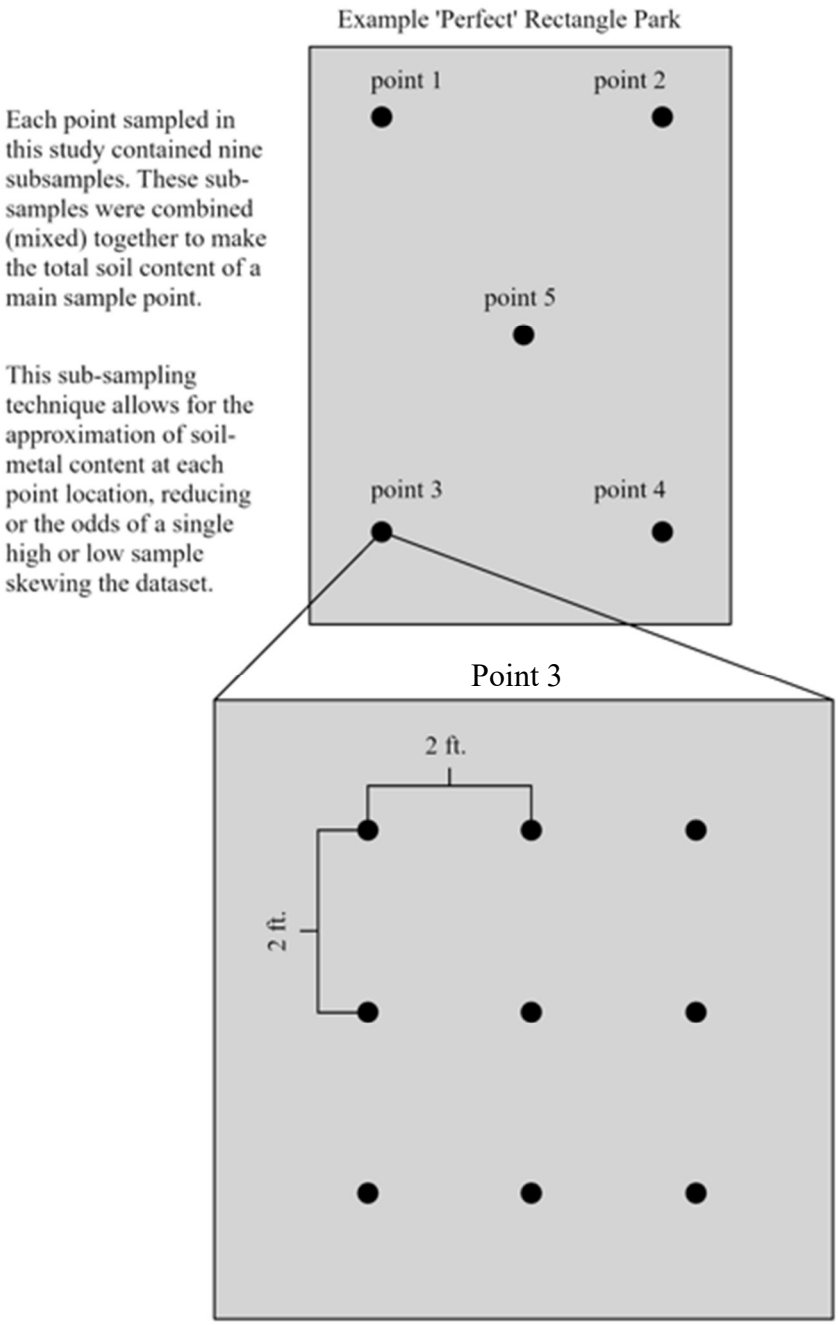

Figure 5. Graphical interpretation of an 'ideal' park. Point 3 sub graphic shows sample protocol at every sample location regardless of park size.

Soil Analysis

At Ball State University, samples were placed in a temperature-controlled freezer. After they were defrosted, samples were air-dried for at least one week at ambient 21℃ temperatures, then placed into a temperature controlled drying oven at 30℃ for 24 hours before sieving through a US Sieve Series No. 10 sieve to achieve homogeneity (EPA, 1996a). Samples were sent to Geochemical Testing – Energy and Environmental Services in Somerset Pennsylvania for analysis. The lab utilized EPA method 3050B for partial digestion of samples for bio-accessible Pb content. The method dissolves all Pb and metal constituents not bound in sulfate matrixes, and as such, is deemed appropriate for a study of this type (EPA, 1996a). The method attempts to mimic conditions found within the human body so as to determine absorbable Pb content. EPA method 6010 was utilized by the lab to analyze the partially digested sample series by ICP-AES, or inductively coupled plasma-atomic emission spectrometry (EPA, 2000). The lab calculated total solids for each sample, which factors out moisture content from the final result (EPA, 1996a).

To determine test accuracy, two duplicate samples were obtained, with results within 8.7% and 3.4% of their counterpart samples. These relatively small variance in result shows acceptable test accuracy at the levels identified in this study.

Pb Screening Levels

The United States EPA as well as Housing and Urban Development (HUD) recommend screening levels of 400 ppm and 1200 ppm for total Pb in residential and industrial soils, respectively (EPA, 2019; HUD, 2012). As the focus of this study is bio-accessible Pb, the California Human and Ecological Risk Office (HERO) stringent guidelines of 80 ppm for bio-available Pb in residential soils was used in place of the HUD guidance for total Pb (Carlisle and

Dowling, 2007; HERO, 2019). California screening values (80ppm bio-available Pb) were determined by the amount of soil-lead found to cause an increase in BLL by one µg/dL (Carlisle and Dowling, 2007). The increase of one µg/dL blood Pb corresponds with a decrease of one IQ point per unit increase in blood Pb (HERO, 2019). The value of 80 ppm was calculated based on a series of parameters contributing to the LeadSpread-8 risk assessment tool, including gastrointestinal uptake, ingestion constants of soil, inhalation constants of dust, breathing rates, surface area of skin, exposure days per week, and a geometric standard deviation value (DTSC, 2011). A gastrointestinal uptake value of 44% of measured soil-lead content is based on a study on rats conducted in 1989, which compared the intestinal absorption of soil-lead against pure Pb acetate (Chaney et al., 1989). The measurement, in short, states that if a soil contains 1000 ppm Pb, then the gastrointestinal system will uptake 44% of the content, making 440 ppm the bio-available quotient. Soil ingestion rates were taken from the EPA's exposure factors handbook, which assumes an average of 100 mg/day of combined soil and dust consumption (EPA, 1997). Inhalation and deposition of dust particulate in the lungs of children is set at 0.192 µg/dL per µg/day, which is based on studies conducted in 1987 comparing adults' respiration rates to that of children; children were found to have a rate of dust deposition 1.6 to 2.7 times higher than that of an adult (EPA, 1986). An average breathing rate of 6.8 m^3/day was used as recommended by the EPA exposure factors handbook (EPA, 1997). Average skin area exposure of 2900 cm^2 for a residential area was calculated in 1998 by the EPA (EPA, 1998). Exposure days per week are assumed to be seven for residential circumstances by the California Department of Toxic Substances Control. The final contributing factor is the geometric standard deviation of 1.6 for blood Pb concentrations (White et al., 1998). The product of the above variables is multiplied by a standard 0.001 value. The result is divided by seven (days) to determine the contribution of soil ingestion to an estimated BLL per each day (DTSC, 2011).

This is then added to soil contact pathways and inhalation pathways to find the 50th percentile estimation of blood Pb (µg/dL). The 90th percentile and up are then calculated from the 50th percentile (DTSC, 2011).

While the LeadSpread-8 model calculates risk levels for bio-available Pb, bio-accessible Pb, as defined by this study is an important factor to address. Applying the 80 ppm value from HERO for bio-available PB over the HUD recommended 400 ppm total Pb allows for a closer examination of problem areas due to the more sensitive bio-available value. Bio-accessibility (or environmental availability) is closer to bio-available levels than total Pb levels.

Iso-concentration Designations

Soil level mapping was first completed by hand on a hardcopy map and then transferred to ArcGIS. The value for this study's iso-concentration line, which is a line used to delineate zones of contamination, was picked based on the conservative California value of 80 ppm. This bio-accessible Pb value was chosen for the iso-concentration boundary.

SPSS

IBM Statistics Package for Social Sciences (SPSS) was primarily used for analyzing interactions between select variables. The program is useful in its capacity to analyze large datasets, as well as easily generating high-quality graphs and high-fidelity numeric output (Wagner, 2019). SPSS allows the user to perform a wide variety of statistical analyses ranging from bivariate plots to multi-variate analyses. Further refinement of the dataset was required prior to its use in statistical study. Most basic analytical methods offered by SPSS necessitate that certain assumptions are met, such as strict limitations in variance between groups, or in this case, no one individual being a subject of multiple tests. If an individual's SID (Security

Identifier) appeared multiple times, it indicated that the person was re-tested. Multiple blood Pb tests occur to monitor an individual's Pb level over time for a reduction due to environmental change, natural attenuation, or chelation therapy. Performing multiple tests on the same individual can skew data especially if the result is a medically induced lower value than the initial test. In all cases of SIDs appearing multiple times, the highest blood Pb result was retained, and all other tests associated with that SID were discarded. To continue analysis, nominal variables required recoding to a new series where the variable was indicated as either exactly the variable (1) or any result other than the variable (0). This recoding process is known as dummy-coding. Male to female variables were replaced with (1) for male and (0) for not male. For nominal variable series, such as race, the dummy-coding created four new variables: White vs. not White, Black vs. not Black, Asian vs. not Asian, and Other vs. not Other; each have a (1) as the variable in question and a (0) for any other result (M. Terwillegar, personal communication, 2019). The determination to use linear regression over one-way ANOVA (Analysis of Variance) lies in linear regression's strength to analyze a continuous independent variable such as percent of a census tract in poverty or percentage of houses in a census tract constructed prior to 1980. The significance threshold for this study was set at 0.05. Preceding analysis, all variables were centered, which entails determining the mean of each variable series, and subtracting it from the series to generate a new variable "C_variable name." The centering process prevents the new interaction variable from affecting the original variable in the statistical model (M. Terwillegar, personal communication, 2019). This centering is important, in that the original variables will not be utilized multiple times during the statistical analysis, thus leaving them unaffected by user actions. Once the recoding and centering was completed, linear regression with BLL (µg/dL) as the dependent variable and all other variables as independent was performed. The analysis included gender, race, age at time of test, season tested, and whether

an individual lived inside or outside the 80 soil iso-concentration zone. Additional analyses tested the interaction of all independent variables. In an effort to produce accurate results, collinearity, which measures whether two variables share the same response and contribute artificial strength to the model, was tested in addition to the linear regression analysis. Variables expressing high potential for collinearity were excluded from the study (M. Terwillegar, personal communication, 2019). Consequently, variables excluded in this manner were statistically insignificant and did not affect the overall variance contribution to BLL.

Results

Blood Lead Levels

Of those tested for blood Pb contamination, more individuals with elevated BLL were located within the southern Muncie footprint, south of the White River, than in the northern Muncie footprint. The mean first-test BLL for the 414 individuals north of the White River was 3.47±4.51 µg/dL, while mean first-test BLLs of 4.53±6.64 µg/dL occurred south of the White River in 468 individuals. BLL varied widely across those tested, with results ranging from 1 µg/dL to 55 µg/dL. At the ≥ 5 µg/dL concentration, south Muncie BLL tests, totaling 107, had a mean BLL of 12.16±10.68 µg/dL, while north Muncie BLL tests (67) had a mean of 9.82±8.57 µg/dL. For the ≥ 10 µg/dL concentration, south Muncie BLL tests (39) had a mean of 22.00±11.80 µg/dL, while northern Muncie tests (21) had a mean of 18.47±11.17 µg/dL. At the highest analyzed level, ≥ 25 µg/dL, the southern Muncie BLL tests (12) held an average of 37.16±9.74 µg/dL while northern Muncie BLL tests (4) had a mean of 37.50±13.07 µg/dL. For a summary of test numbers, see Table 1. Study site maps outlining blood Pb concentrations ≥ 0, ≥ 10, and ≥ 25 µg/dL are included as Figures 6, 7, and 8 respectfully. Comparing both number of blood Pb samples and relative BLL yielded no noteworthy districts.

Seasonal distribution of testing was consistent; 328 tests were conducted in spring with a mean test result of 5.00±7.48 µg/dL, while 349 tests were conducted in summer with a mean of result of 4.33±5.57 µg/dL. Fall and winter conducted 346 and 344 tests with 4.64±5.99 µg/dL and 3.64±4.30 µg/dL respectively. Table 2 contains summarized seasonal statistics. Maps including poverty and housing construction data are provided in Figure 9 and Figure 10 respectively. The data from these maps were incorporated into later statistical analysis.

Table 1. Distribution of blood Pb averages with respect to the White River.

µg/dL	Orientation from White River	Total Individuals	Mean BLL (µg/dL)
All BLL in area	North	414	3.47±4.51
	South	468	4.53±6.64
≥ 5	North	67	9.82±8.57
	South	107	12.16±10.68
≥ 10	North	21	18.47±11.17
	South	41	22.00±11.80
≥ 25	North	4	37.50±13.07
	South	12	37.16±9.74

Table 2. Seasonal testing statistics

Season	Total Tests	Mean (µg/dL)
Spring (Mar-May)	328	5.00±7.48
Summer (Jun-Aug)	349	4.33±5.57
Fall (Sept-Nov)	346	4.64±5.99
Winter (Dec-Feb)	344	3.64±4.30

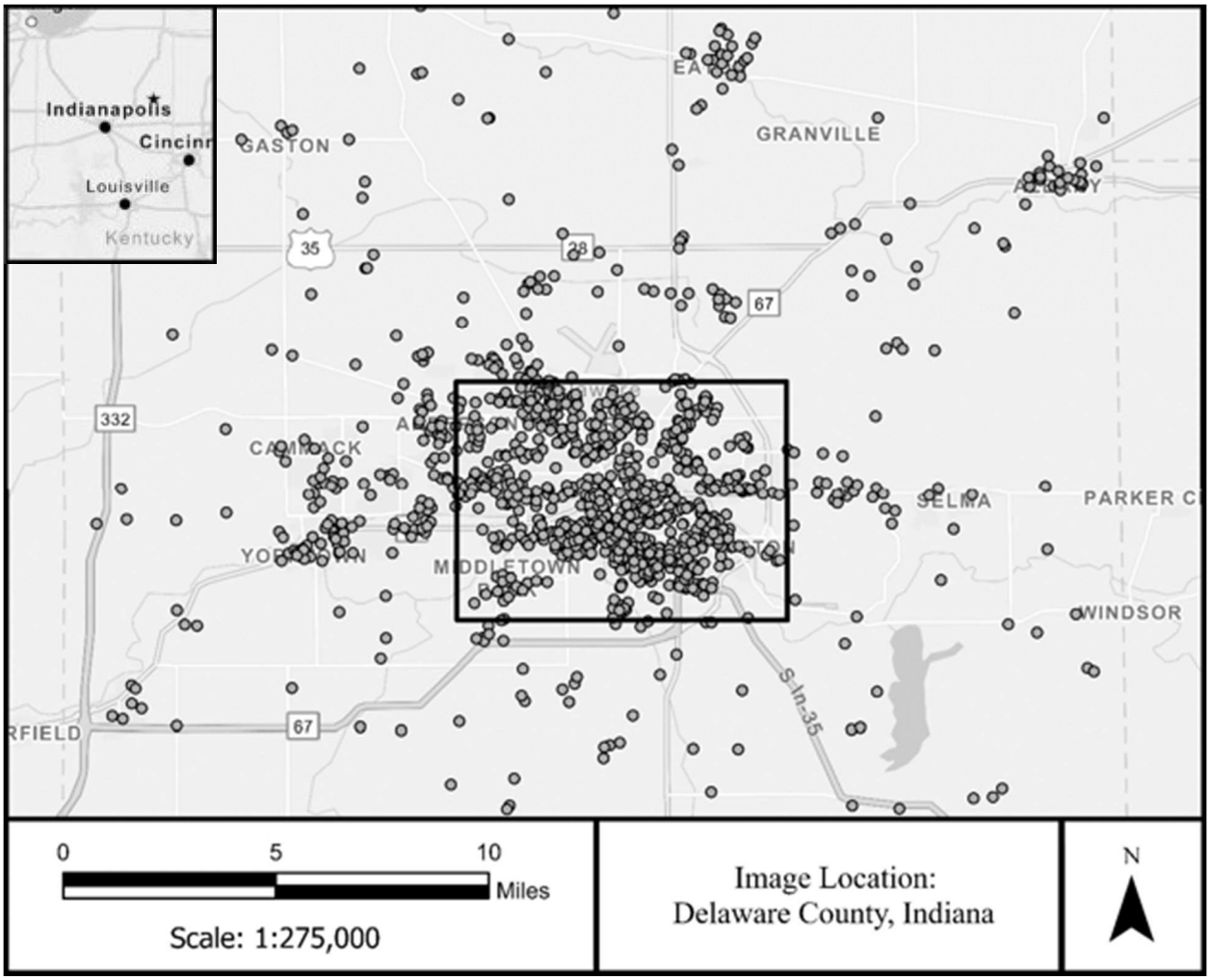

Figure 6. Map of study area displaying BLL greater than zero µg/dL; the black box outlines the primary area of focus encompassing Muncie census tracts. Inset map indicates location of Muncie (star) in relation to rest of Indiana.

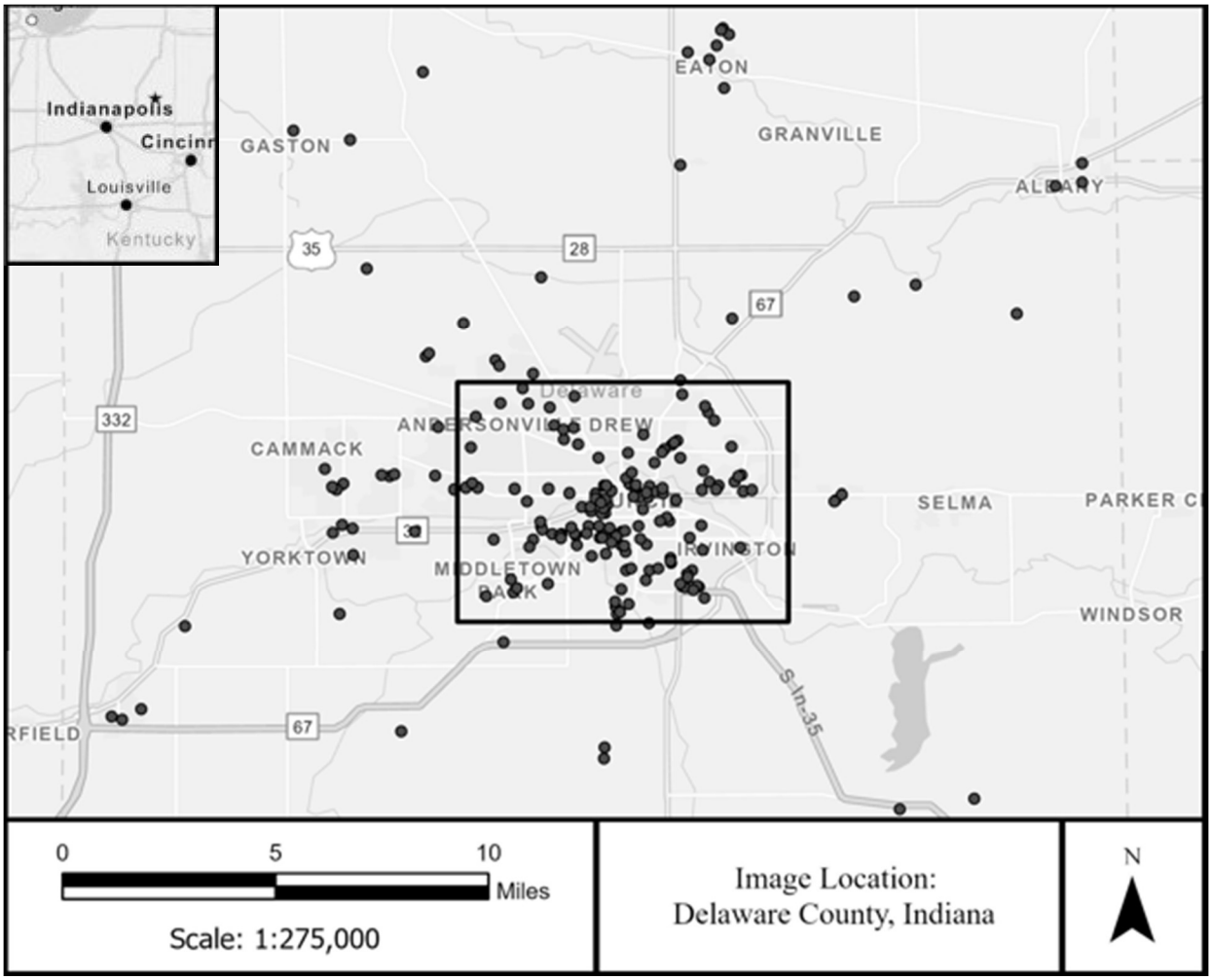

Figure 7. Map of study area displaying BLL greater than or equal to 5 µg/dL; the black box outlines the primary area of focus encompassing Muncie census tracts. Inset map indicates location of Muncie (star) in relation to rest of Indiana.

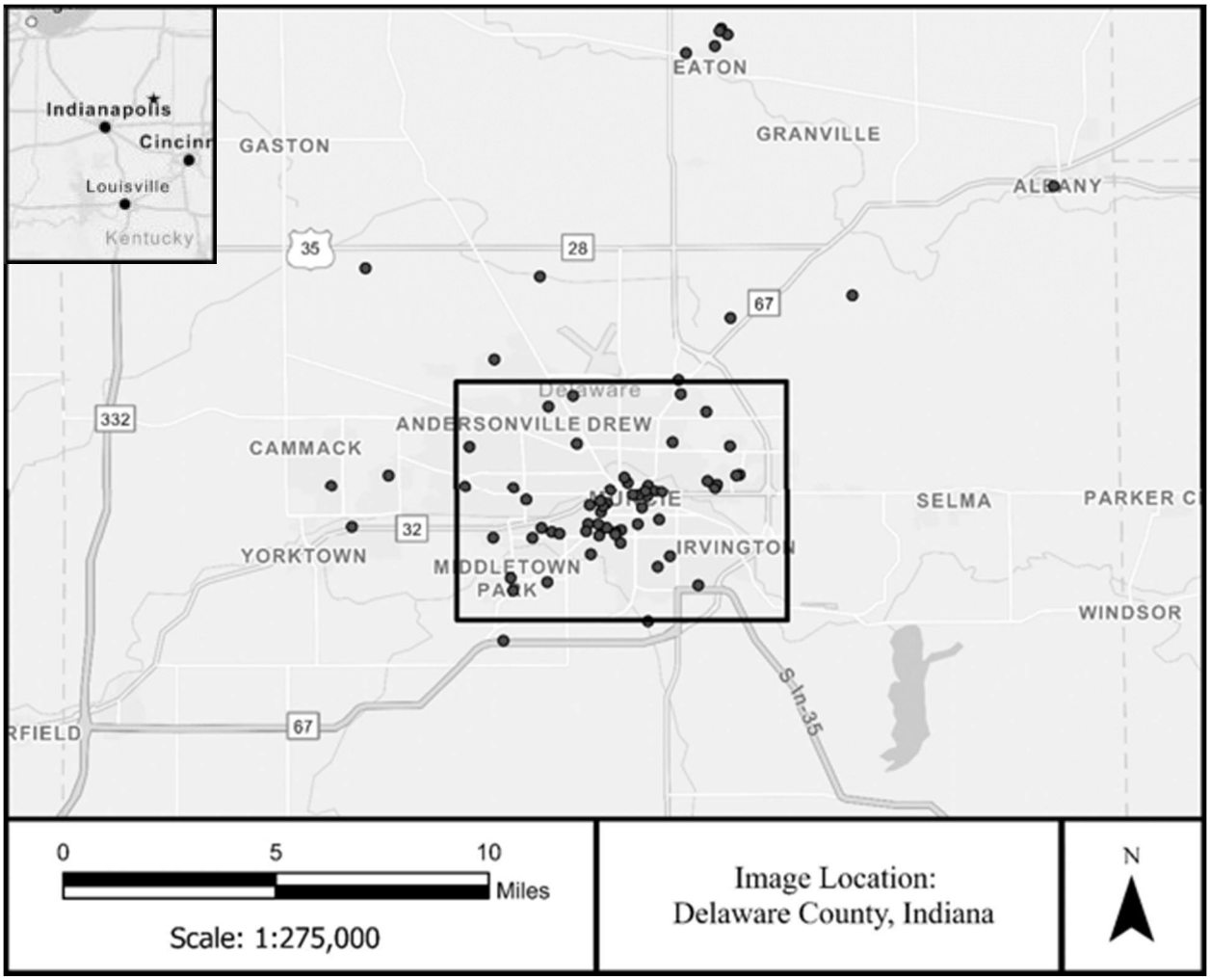

Figure 8. Map of study area displaying BLL greater than or equal to 10 µg/dL; the black box outlines the primary area of focus encompassing Muncie census tracts. Inset map indicates location of Muncie (star) in relation to rest of Indiana.

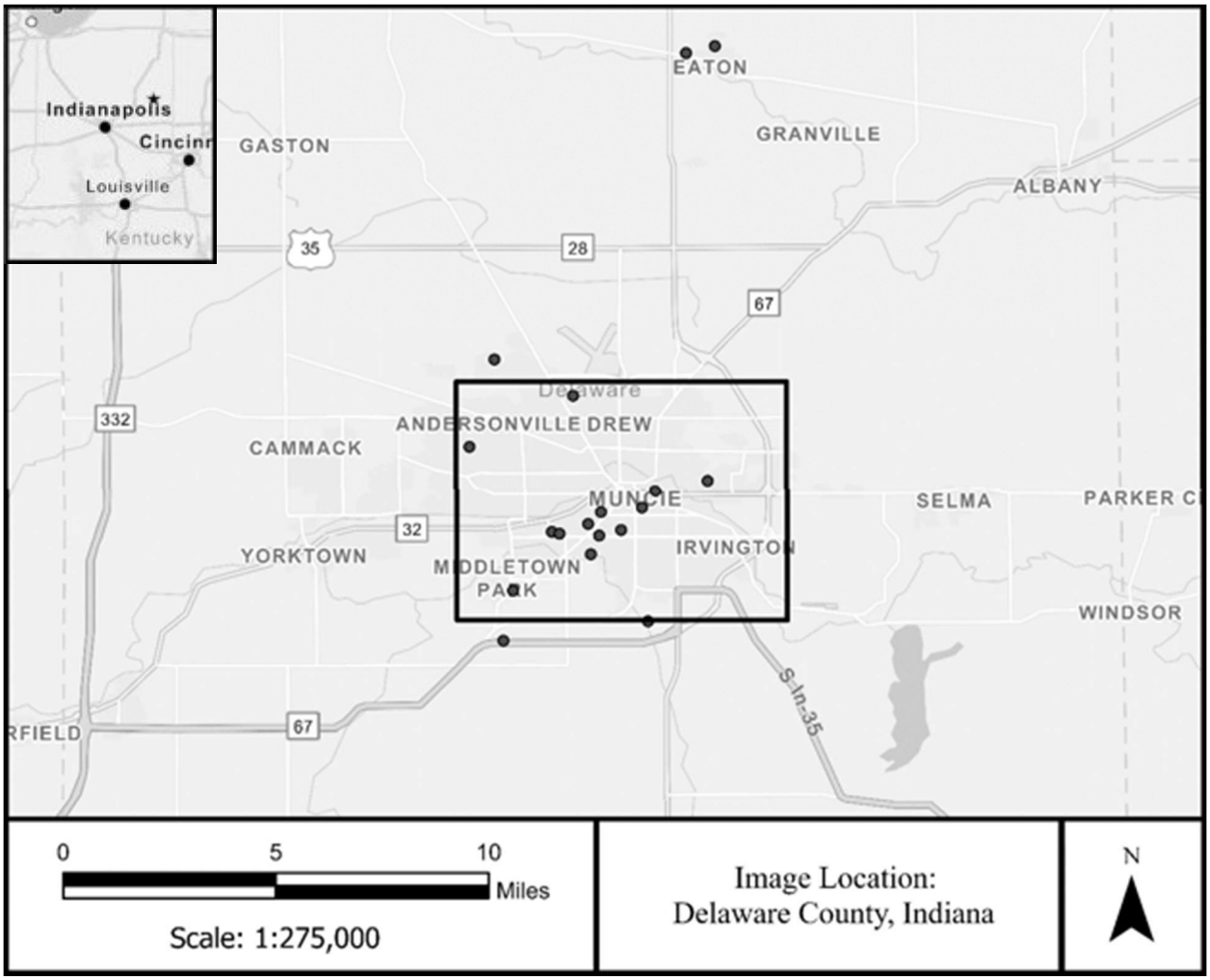

Figure 9. Map of study area displaying BLL greater than or equal to 25 µg/dL; the black box outlines the primary area of focus encompassing Muncie census tracts. Inset map indicates location of Muncie (star) in relation to rest of Indiana.

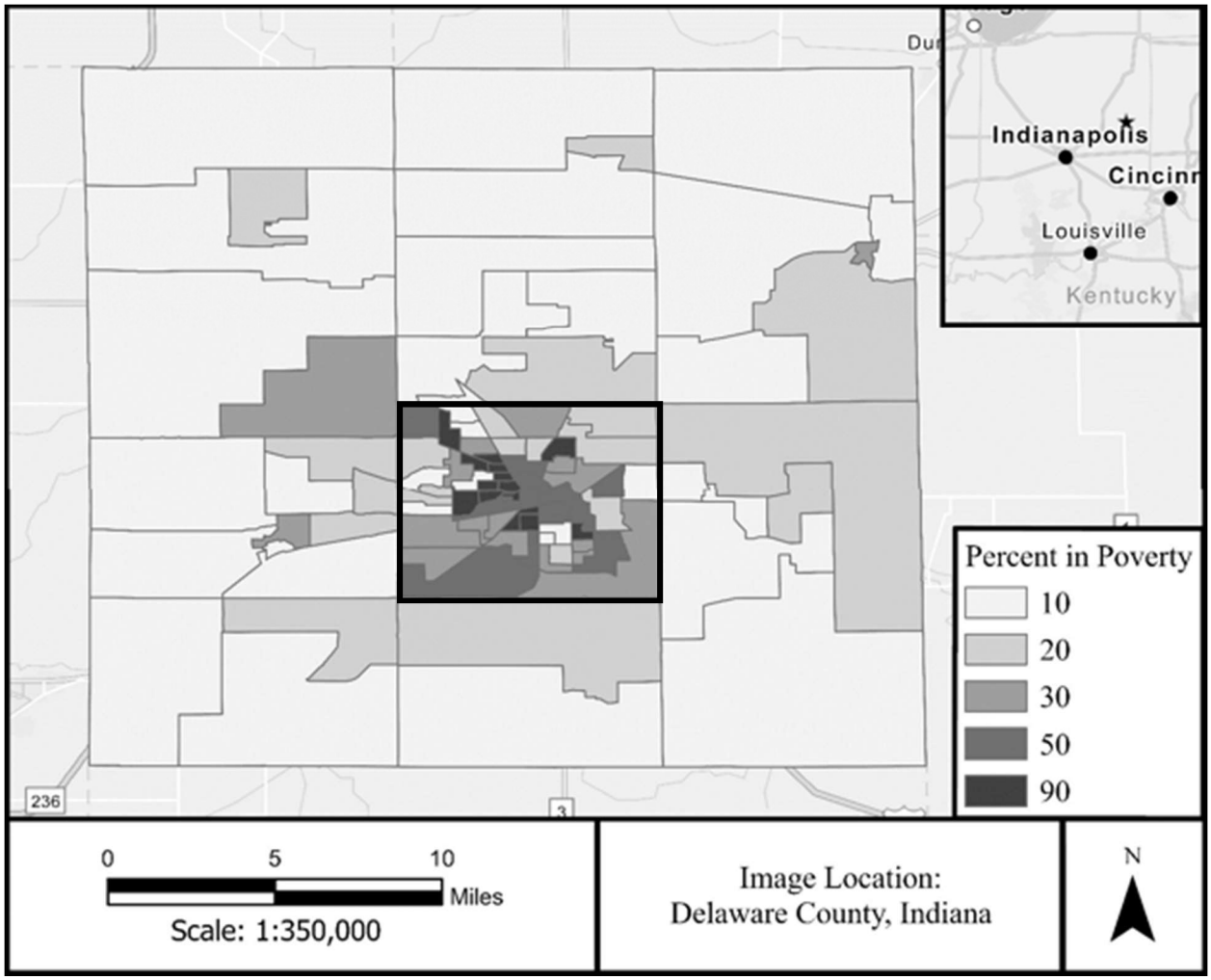

Figure 10. Map of Muncie poverty at the census tract level. Data were collected by American FactFinder for 2017 poverty levels. The inset map shows the study area of Muncie (star) with relation to Indiana (Bureau, 2017). The black (bold) outlined box indicates the primary study area.

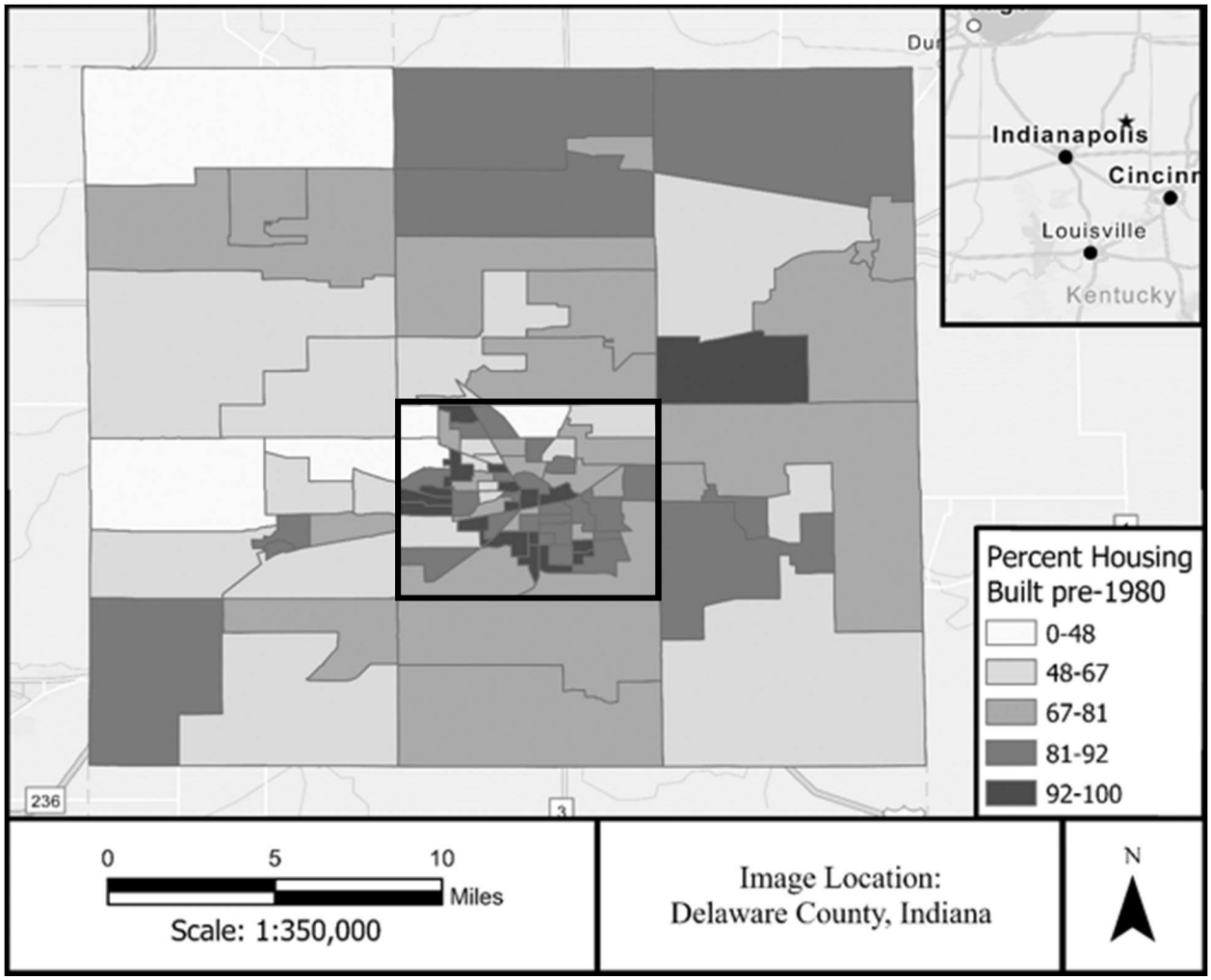

Figure 11. Map of Muncie housing constructed prior to 1980 at the census tract level. Data were collected by American FactFinder for the year 2014 (Bureau, 2014). Inset map shows study area of Muncie (star) with relation to the rest of Indiana. The black (bold) outlined box indicates the primary study area.

Soil Metals Levels

The 88 soil samples, including two blind duplicates, and two samples collected to determine background levels were analyzed by EPA method 3050B (EPA, 1996a). Samples were analyzed for cadmium, chromium, copper, lead, and nickel. See Table 3 for a summary of soil analytical data. Of all analyzed samples, 22 locations were above the 80 ppm (bio-accessible) soil Pb level recommended by California HERO.

Table 3. Analytical results of soil samples collected Fall 2018 (F18) through Summer 2019 (SUM19); all values are expressed in parts per million (ppm) and are considered bio-accessible based on the EPA 3050B analysis. Sample naming scheme is as follows: Season and year sampled_Park name_Sample number.

Sample Name	Cadmium	Chromium	Copper	Lead	Nickel
Altshire Park					
F18_ALTSHPRK_001	5.0	12.2	14.6	19.9	11.4
F18_ALTSHPRK_002	5.0	13.7	16.1	42.1	14.1
F18_ALTSHPRK_003	5.0	19.4	25.7	36.6	22.2
F18_ALTSHPRK_004	5.0	16.4	19	28.7	25.1
F18_ALTSHPRK_005	5.0	18.3	21	32.1	19.1
Ball Corp Park					
F18_BALLCOPRK_001	5.0	13.4	26.1	60	16.2
F18_BALLCOPRK_002	5.0	13.1	26.2	47.4	14.9
F18_BALLCOPRK_003	5.0	11.2	22.2	34.6	14.2
S18_BALLCOPRK_004	5.0	13	39.4	33.8	18.4
S18_BALLCOPRK_005	5.0	13.7	23.8	35	15.9
Clifton Wallace Park					
F18_CLIWALPRK_001	5.0	15.5	22.6	143	19.8
F18_CLIWALPRK_002	5.0	10.8	16	62.9	14.5
F18_CLIFWALPRK_002_Dup	5.0	11.4	14	68.9	15.2
F18_CLIWALPRK_003	5.0	15.4	36	366	20.4
Cooley Park					
F18_COOLPRK_001	5.0	10.6	16.8	31.7	13.1
F18_COOLPRK_002	5.0	11.7	16.9	22.1	15.8
F18_COOLPRK_003	5.0	11.7	26.8	26.3	15.8
F18_COOLPRK_004	5.0	11.4	17.4	33.7	14.4
F18_COOLPRK_005	5.0	18.4	25.4	33.7	21.3

Table 3 (cont). Analytical results of soil samples.

Sample Name	Cadmium	Chromium	Copper	Lead	Nickel
DIY Point					
F18_DIYPT_001	5.0	14.6	26.1	103	17.1
Fireman's Memorial Park					
F18_FIREPRK_001	5.0	12.9	16.6	26.4	14
Friend's Memorial Church					
F18_FRNDSMEMCHRH_001	5.0	15.9	20.9	142	15.2
Gilbert Park					
F18_GLBTPRK_001	5.0	16.2	31.9	205	19.2
Ginn Woods					
F18_GNWDS_001	5.0	14.1	16.2	18	18
F18_GNWDS_002	5.0	23.8	35.3	41.2	31.2
Heekin Park					
F18_HKNPRK_001	5.0	13.5	17.4	57.7	13.9
F18_HKNPRK_002	5.0	11.8	20.8	68.7	14.5
F18_HKNPRK_003	5.0	11.2	15.7	47.4	11
F18_HKNPRK_004	5.0	18.3	29	140	21
Cowing Park					
F18_COWPRK_001	5.0	15.2	20	28.2	20.1
F18_COWPRK_002	5.0	17.1	21.4	47.1	21.5
F18_COWPRK_003	5.0	13.4	16.4	40.4	13.5
F18_COWPRK_004	5.0	12.1	16.9	30.5	11.9
F18_COWPRK_005	5.0	19.6	23	32.4	26.3
Emerson Dog Park					
F18_EMRNPRK_001	5.0	15.1	31.7	45.1	19.7
F18_EMRNPRK_003	5.0	14.6	29.5	50.5	18.7
F18_EMRNPRK_004	5.0	14.4	22.2	56.1	21.2
F18_EMRNPRK_005	5.0	14.9	25.9	90.4	22.1

Table 3 (cont). Analytical results of soil samples

Sample Name	Cadmium	Chromium	Copper	Lead	Nickel
Hazlewood Christian Church					
F18_MANSION_001	5.0	16.6	21.5	78.5	16.9
Mcculloch Park					
F18_MCLCPRK_001	5.0	15.8	21.8	35.1	17.8
F18_MCLCPRK_002	5.0	13.8	14.2	53.4	12.5
F18_MCLCPRK_003	5.0	16.5	19.8	43.8	20.6
F18_MCLCPRK_004	5.0	24.3	22	171	20.8
F18_MCLCPRK_005	5.0	14.1	24.1	91.2	17.6
Millennium Park					
F18_MILLPRK_001	5.0	14.3	20.2	37.1	19
F18_MILLPRK_002	5.0	12.1	21.7	35.6	18
F18_MILLPRK_003	5.0	11.9	19.3	36.3	15.9
Point Northwest of Manual Transmission of Muncie Plant					
F18_NWGRPLT_001	5.0	32.3	64.2	623	23.6
Plum Park					
F18_PLMMPRK_001	5.0	14.3	26.6	117	15.9
Riverbend Park					
F18_RBPRK_001	5.0	15.5	19.5	18.8	17.9
Rose Park					
F18_ROSEPRK_001	5.0	15	17.1	70.8	15.1
F18_ROSEPRK_002	5.0	14.2	19.8	109	18.1
F18_ROSEPRK_003	5.0	9.2	16.9	74.4	12.1
F18_ROSEPRK_004	5.0	15.6	18.8	84.7	18.4
F18_ROSEPRK_005	5.0	13.6	16.8	51.9	17.2
Point Southeast of Manual Transmission of Muncie Plant					
F18_SEGRPLT_001	5.0	34.2	134	457	27.6

Table 3 (cont). Analytical results of soil samples.

Sample Name	Cadmium	Chromium	Copper	Lead	Nickel
Thomas Park					
F18_THMPRK_001	5.0	11.6	13.7	46.3	11.6
F18_THMPRK_002	5.0	17.1	20.4	34.4	21.1
F18_THMPRK_003	5.0	11.6	12.7	45.4	11.6
F18_THMPRK_004	5.0	14	15.4	76.4	16.8
F18_THMPRK_005	5.0	13	19.8	105	16.8
Tuhey Park					
F18_TUHPRK_001	5.0	16.1	27.2	134	21.8
F18_TUHPRK_002	5.0	11.5	23.7	154	15.3
F18_TUHPRK_003	5.0	17.7	28.1	122	21.6
F18_TUHPRK_004	5.0	11.8	15.8	33.2	15.7
F18_TUHPRK_005	5.0	11.5	21.6	57.6	15.7
Washington Park					
F18_WASHPRK_001	5.0	12	17.4	38.2	14.8
F18_WASHPRK_002	5.0	13	15.9	44.2	14.2
Westside Park					
F18_WESTPRK_001	5.0	12.3	17.4	68.8	15.3
F18_WESTPRK_002	5.0	14.1	22	62.6	18.6
F18_WESTPRK_003	5.0	14	27.1	62.7	16.9
F18_WESTPRK_004	5.0	15.8	41.8	300	18.8
F18_WESTPRK_005	5.0	23	39.5	377	23.9
White River Park					
F18_WHTRV_001	5.0	10.4	13.1	47.4	9
F18_WHTRV_002	5.0	19.5	18.9	82	14
F18_WHTRV_004	5.0	14.8	15.9	67.7	12.6
F18_WHTRV_004_Dup	5.0	15.3	17.2	70.5	13.4
F18_WHTRV_005	5.0	17.4	20.6	52	17
F18_WHTRV_006	5.0	14.3	21.9	77.1	15.7

Table 3 (cont). Analytical results of soil samples.

Sample Name	Cadmium	Chromium	Copper	Lead	Nickel
Dutro-Ernst Woods					
SUM19_DEWDS_001	5.0	12.2	14.4	35.1	15.5
SUM19_DEWDS_002	5.0	15.2	21.5	70	21.8
SUM19_DEWDS_003	5.0	14.7	25.7	57.8	25.5
SUM19_DEWDS_004	5.0	14.4	19	25.1	25.9
SUM19_DEWDS_005	5.0	15.2	16.6	36.7	17.4

It should be noted that while the EPA reports method 3050B as a total metal analysis, the method is defined by searching for environmentally available metals (bio-accessible). All metals analyzed within this study are considered bio-accessible. The EPA screening table utilizes a variety of methods; however, it utilizes method 3050B for all metals discussed in this report. Correlations between each of the tested metals produced relatively strong positive results between copper and chromium (0.72), and between Pb and copper (0.71). Pb and chromium (0.65) have a positive correlation slightly weaker than that of the other two metal pairs (Table 4).

Table 4. Correlations between metal constituent levels in soil samples using the Pearson Product-Moment Correlation Coefficient method in Excel.

Parameter	Chromium	Copper	Lead	Nickel
Chromium	1			
Copper	0.723626	1		
Lead	0.650208	0.706372	1	
Nickel	0.599557	0.497256	0.313831	1

See Figures 12 through 21 for a series of graphs examining metal concentrations based on the full dataset, census tracts, and comparisons between the various metals analyzed. All analyzed metals with the exception of Pb are at least one order of magnitude below the deemed safe remediation levels proposed by the EPA (EPA, 2019). It should be noted that the sample points (NW and SE Gear plant) were excluded due to their status as an outlier in the dataset.

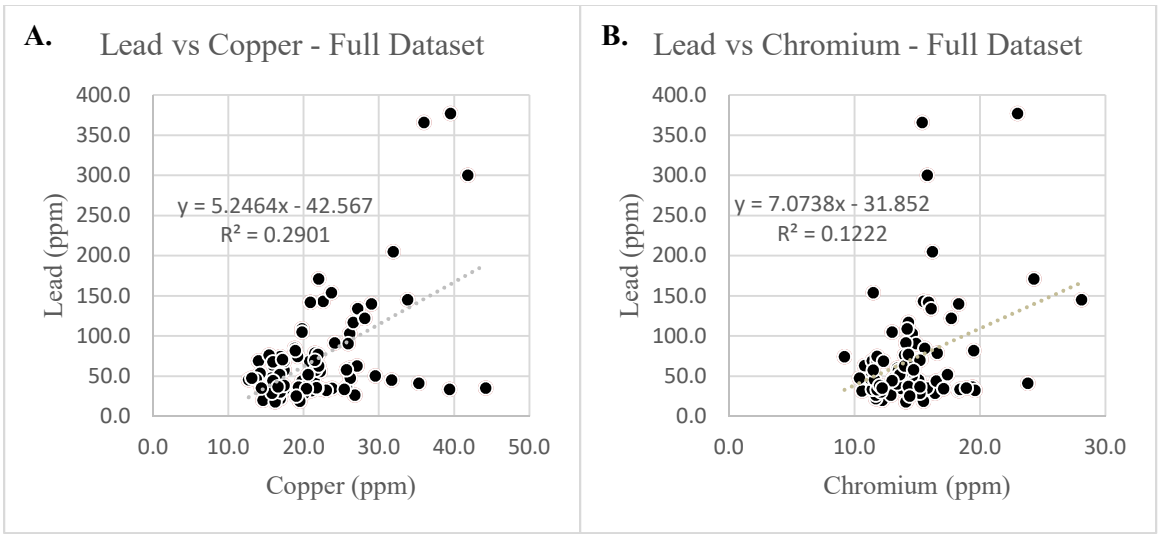

Figure 12. Relations between lead and copper (A) and Lead and Chromium (B).

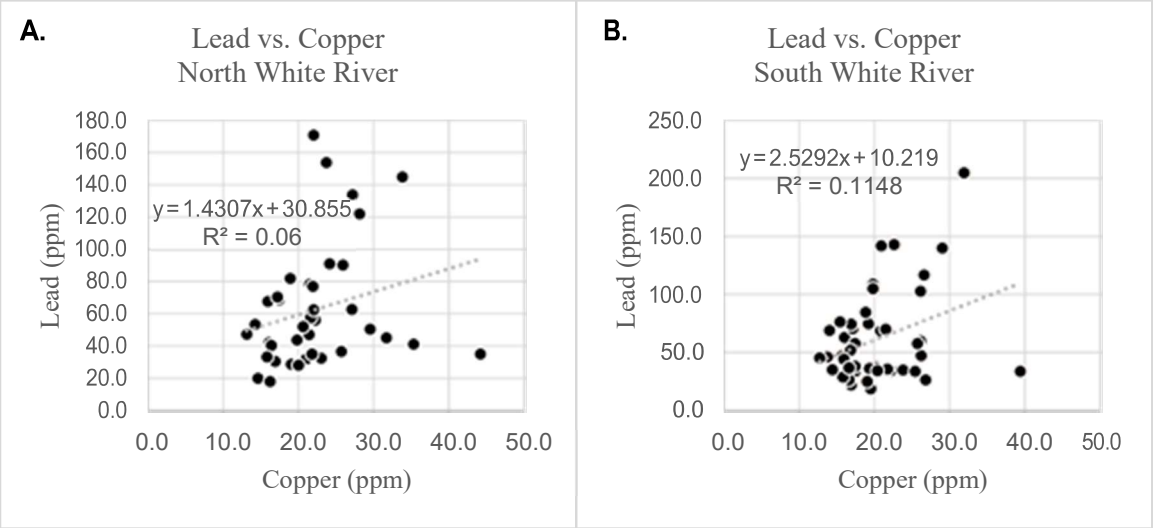

Figure 13. Lead vs. copper levels north (A) and south (B) of the White River in Muncie, Indiana.

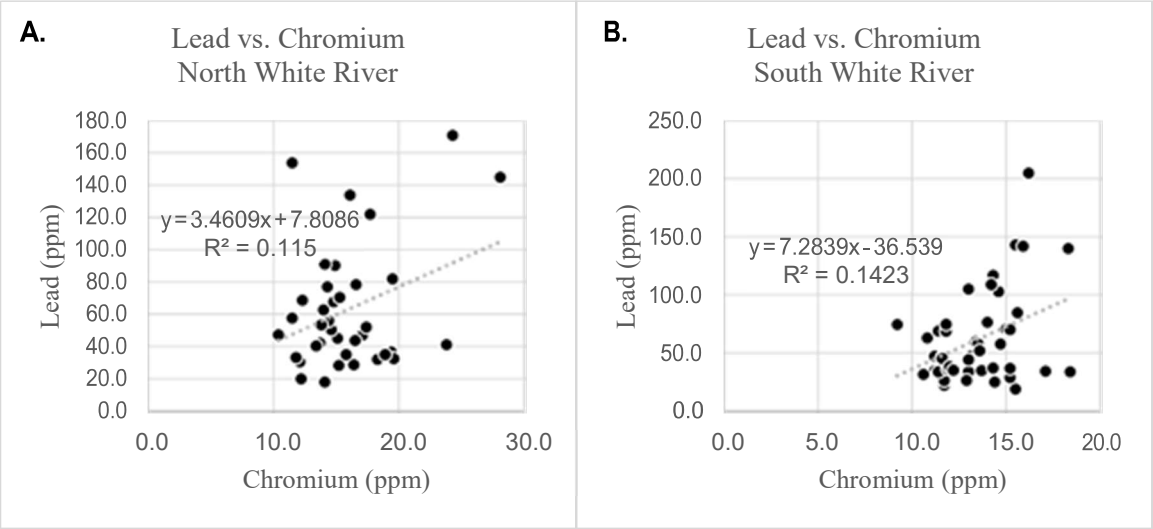

Figure 14. Lead vs. chromium levels north (A) and south (B) of the White River in Muncie, Indiana.

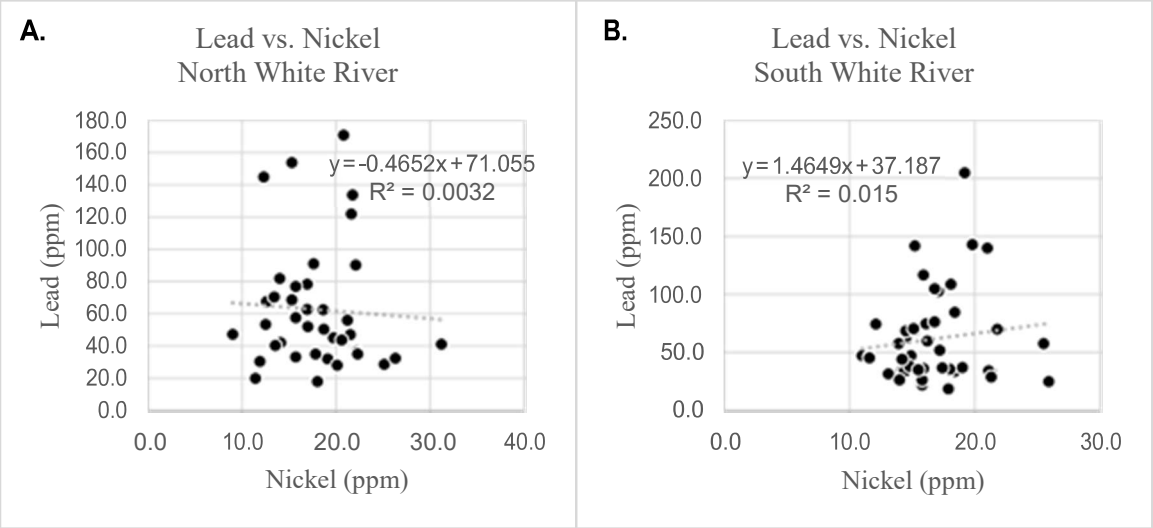

Figure 15. Lead vs. nickel levels north (A) and south (B) of the White River in Muncie, Indiana.

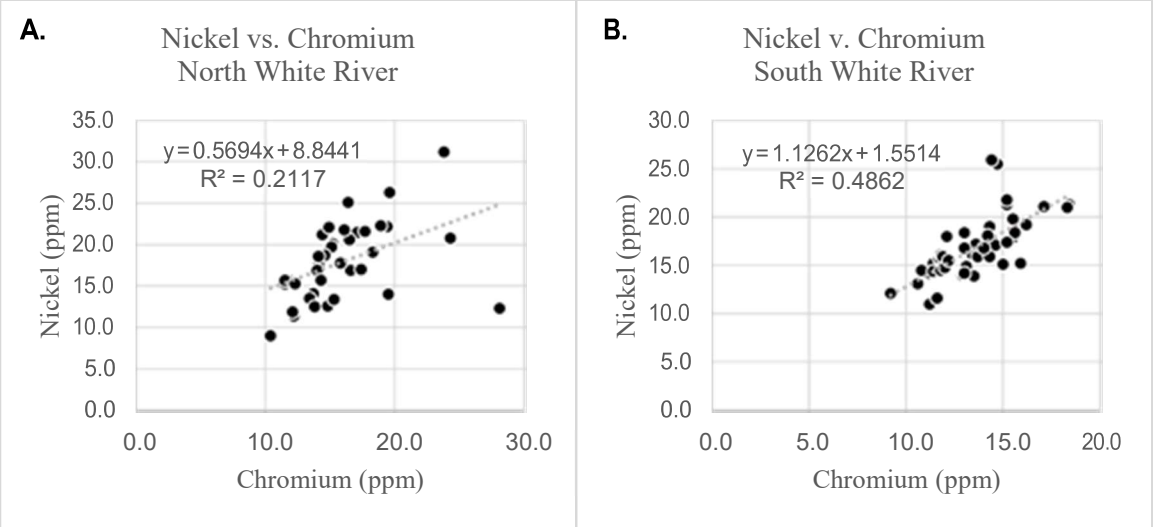

Figure 16. Nickel vs. chromium levels north (A) and south (B) of the White River in Muncie, Indiana.

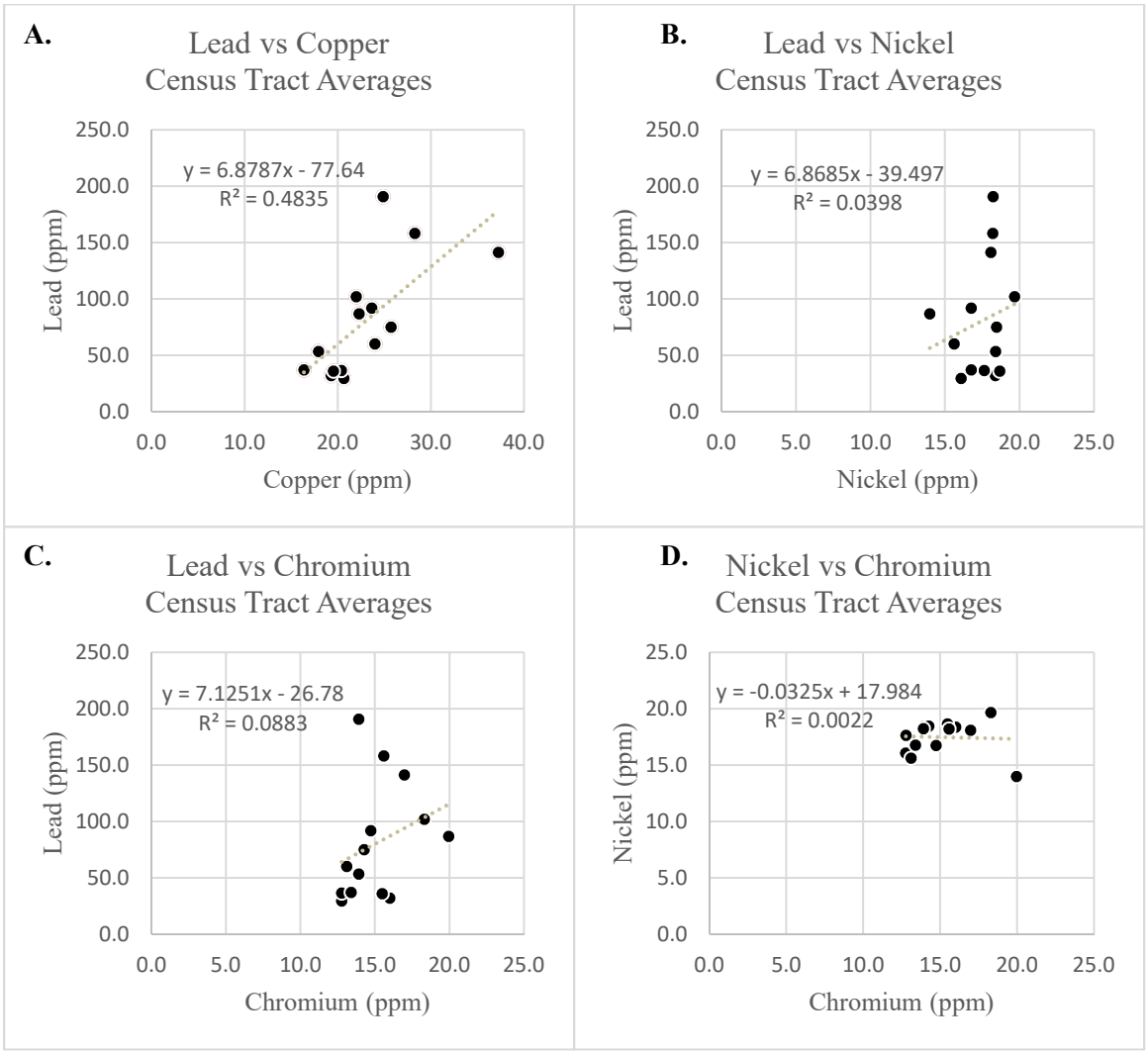

Figure 17. Relations between lead and copper (A), lead and nickel (B), Lead and Chromium (C), and Nickel and Chromium (D) among the Muncie, Indiana census tracts.

Spatial analysis of select sites (with at least 2 samples taken) was performed with respect to the graphed values. Stand-alone graphs with associated maps are included as Figures 18 and 19. On first examination, several clusters are identifiable, however not only is inter-park variation apparent in the following Figures, but the distribution of contaminant levels vary across large distances. Figures 18 and 19 show Clifton Wallace Park as containing both high and low lead

levels, as well as other parks containing points in both clusters. The point distribution across clusters prevents the identification of unique contaminant plumes.

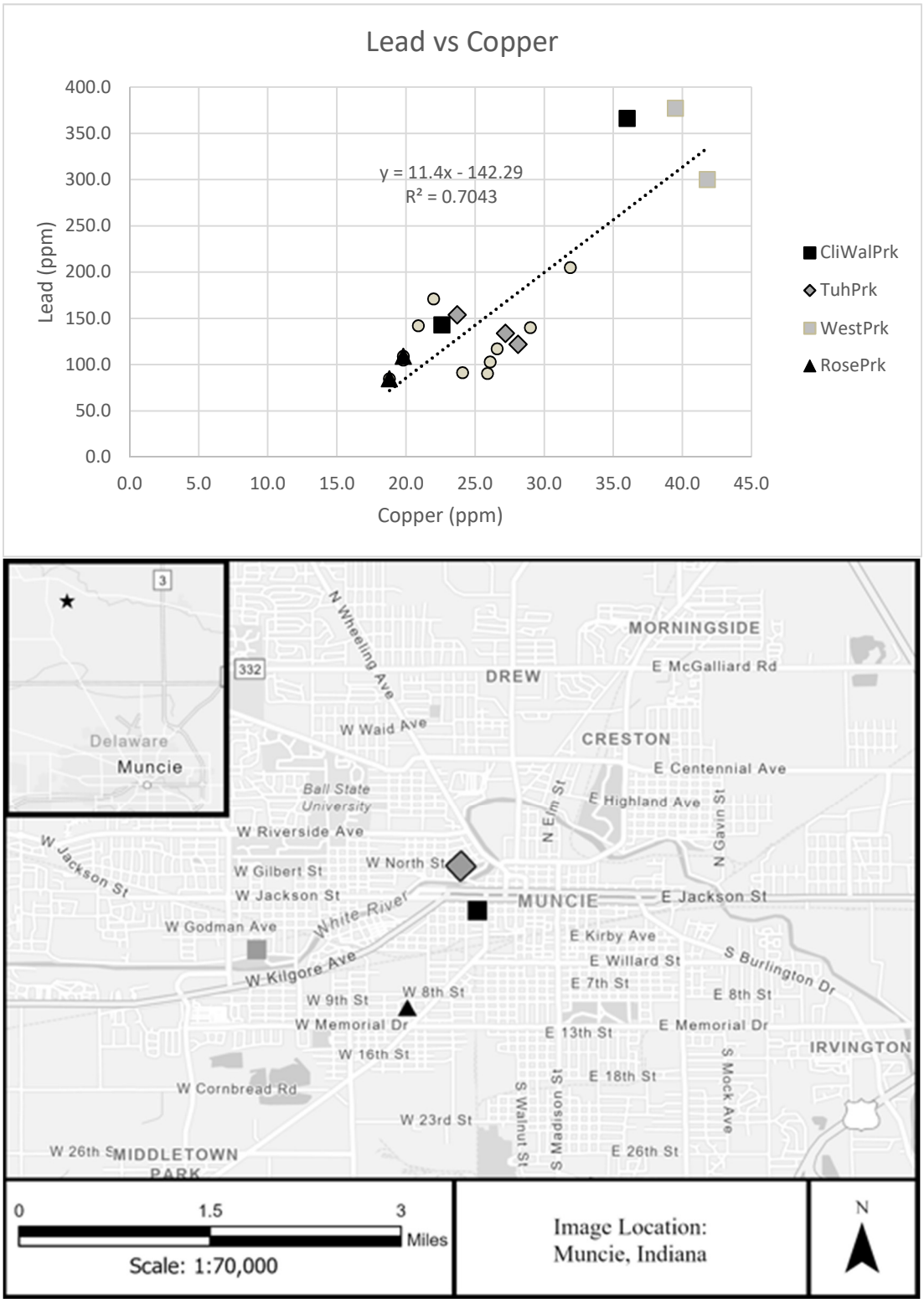

Figure 18. Clustering effects and their corresponding locations on the map with relation to Muncie for lead and copper. Black outlined circles indicate individual sample point values.

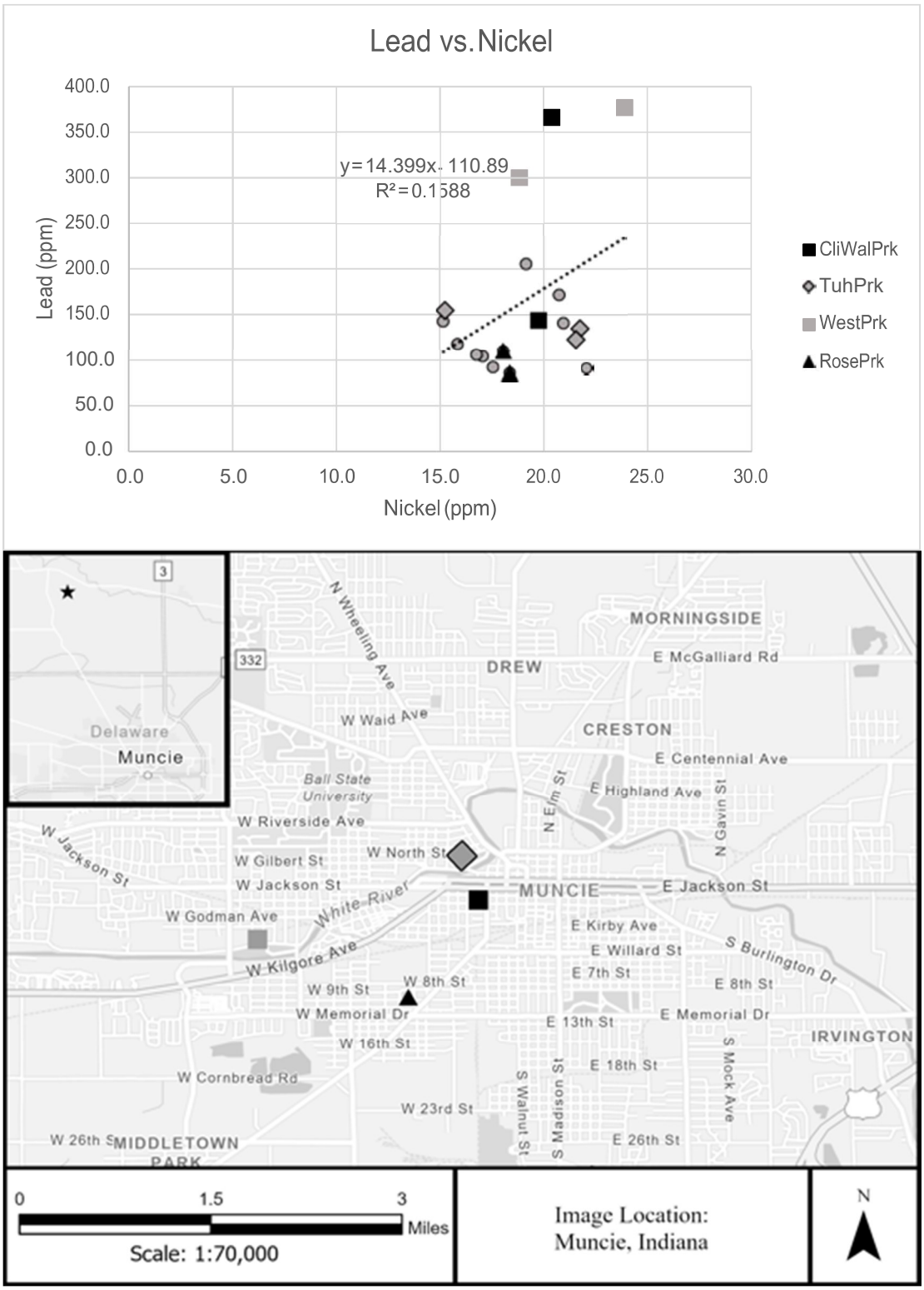

Figure 19. Clustering effects and their corresponding locations on the map with relation to Muncie for lead and nickel. Black outlined circles indicate individual sample point values.

An additional spatial analytical method was utilized for the Muncie census tracts average levels. Figure 20 is included with potential clusters identified on its associated map.

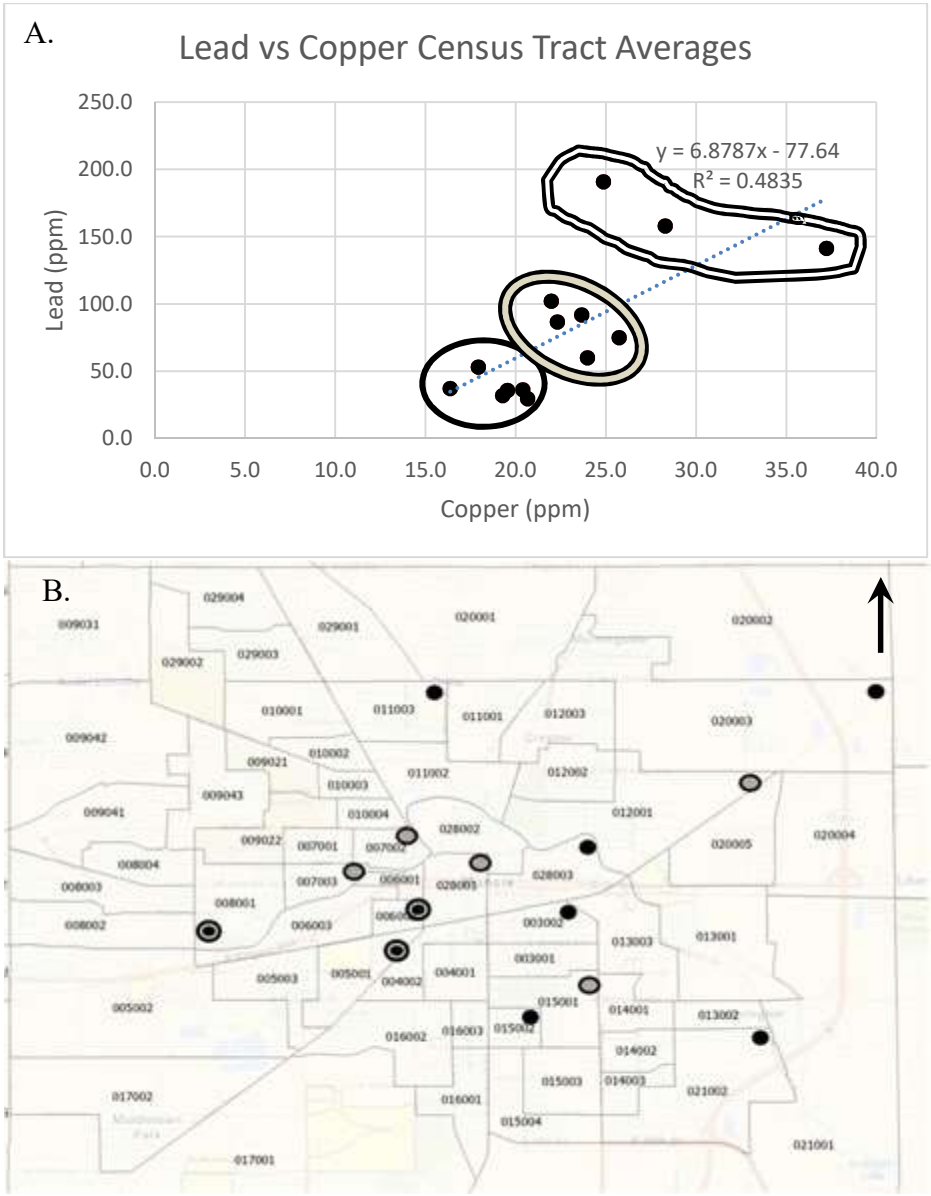

Figure 20. A) outlines clusters in census tract averages for lead vs. copper graphing. Each of the outline styles corresponds to the associated point style in B. B) maps out the points from graph A, showing the distribution patterns of the points from the clusters. The solid black circled cluster corresponds with the solid black points, grey with black outline with black outlined grey points, and double outline with the bullseyes.

Bio-accessible soil data were entered into a GIS database and used to generate an iso-concentration map of Pb levels across Muncie. The map utilized an iso-concentration line based on soil analytical results of 80 ppm (Figure 21). The iso- concentration line forms a concentric polygon over the now defunct Manual Transmission of Muncie facility, spreading primarily in a west-southwest to east-northeast pattern. Soil-lead levels tend to decrease with distance from the former transmission plant. No elevated anomalous samples were located outside the 80 ppm iso-concentration line. Figure 22 outlines brownfield and other former industrial centers with respect to the iso-concentration mapping.

Table 5 summarizes site conditions at each of the locations described in Figure 22. An additional map, Figure 23, combines the Figures 21 and 22 for better comparative ease. The average bio- accessible Pb concentration of samples taken for background sampling was 29.6±16.4 ppm, which falls 1.1 ppm below the total Pb mean of 30.7±54.4 ppm for samples obtained by the EPA for the state of Indiana (EPA, 2018). While the EPA test methodology is for total Pb, it serves as an indicator; the EPA study shows the total soil lead content is low in background measurements. The study area mean bio-accessible soil Pb concentration is 84.01±96.82 ppm, which is more than double both the Indiana state background total Pb average and this study's measured bio-accessible background average. Two duplicate samples were collected from Clifton Wallace and White River parks and Pb levels were within 8.7% and 3.4% of the original sample result respectively. These results are acceptable as intra-sample variation will still occur as true homogeneity will not occur on this scale. Additional analysis of the 80-iso concentration zone and its influence on metal levels was conducted. Graphs outlining this influence are included as Figures 24 through 28.

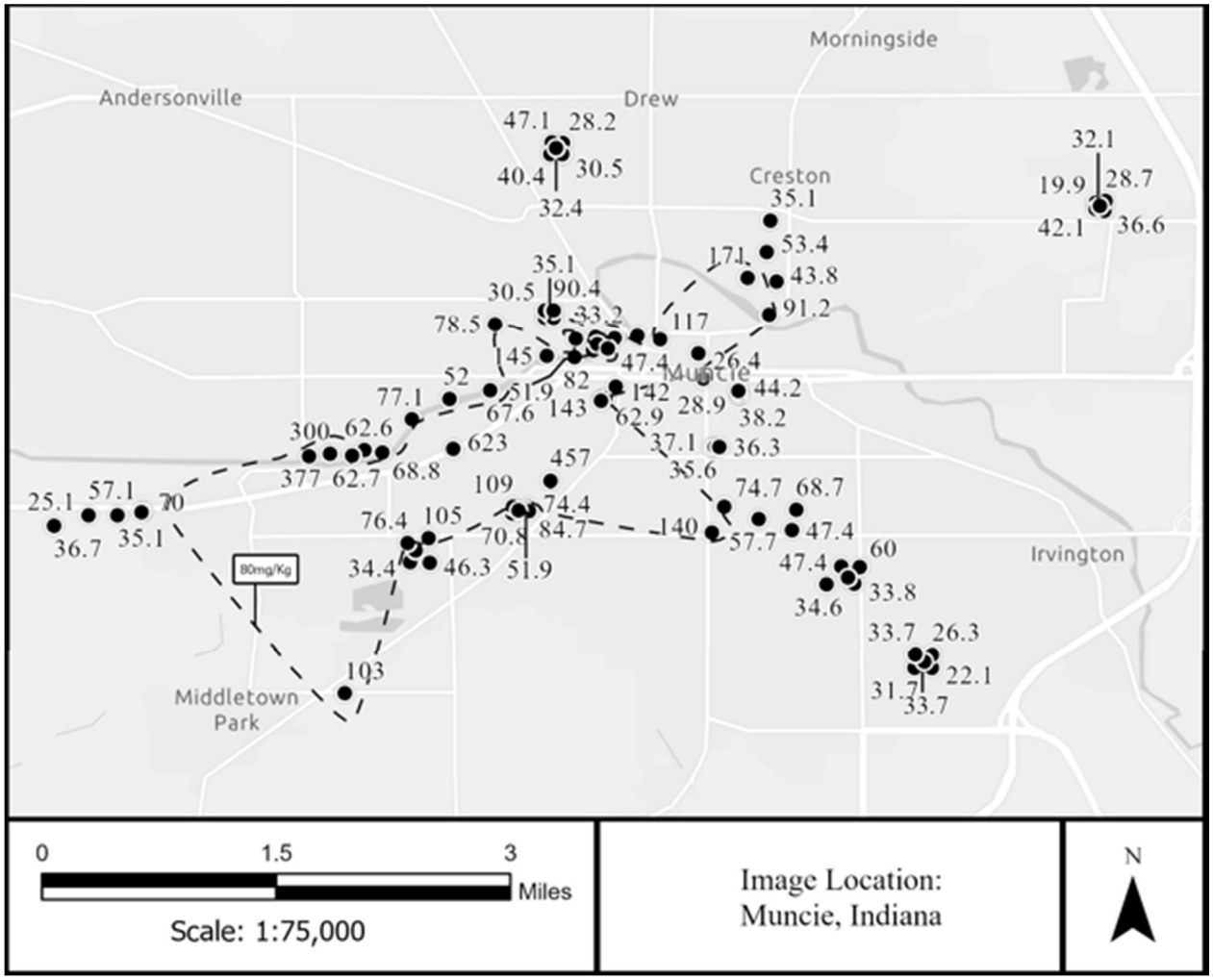

Figure 21. Map including sample analytical data: Iso-concentration lines overlain to show extents of soil-lead distribution; 80 mg/kg is California residential soil-lead screening level for bio-accessible Pb (HERO, 2019).

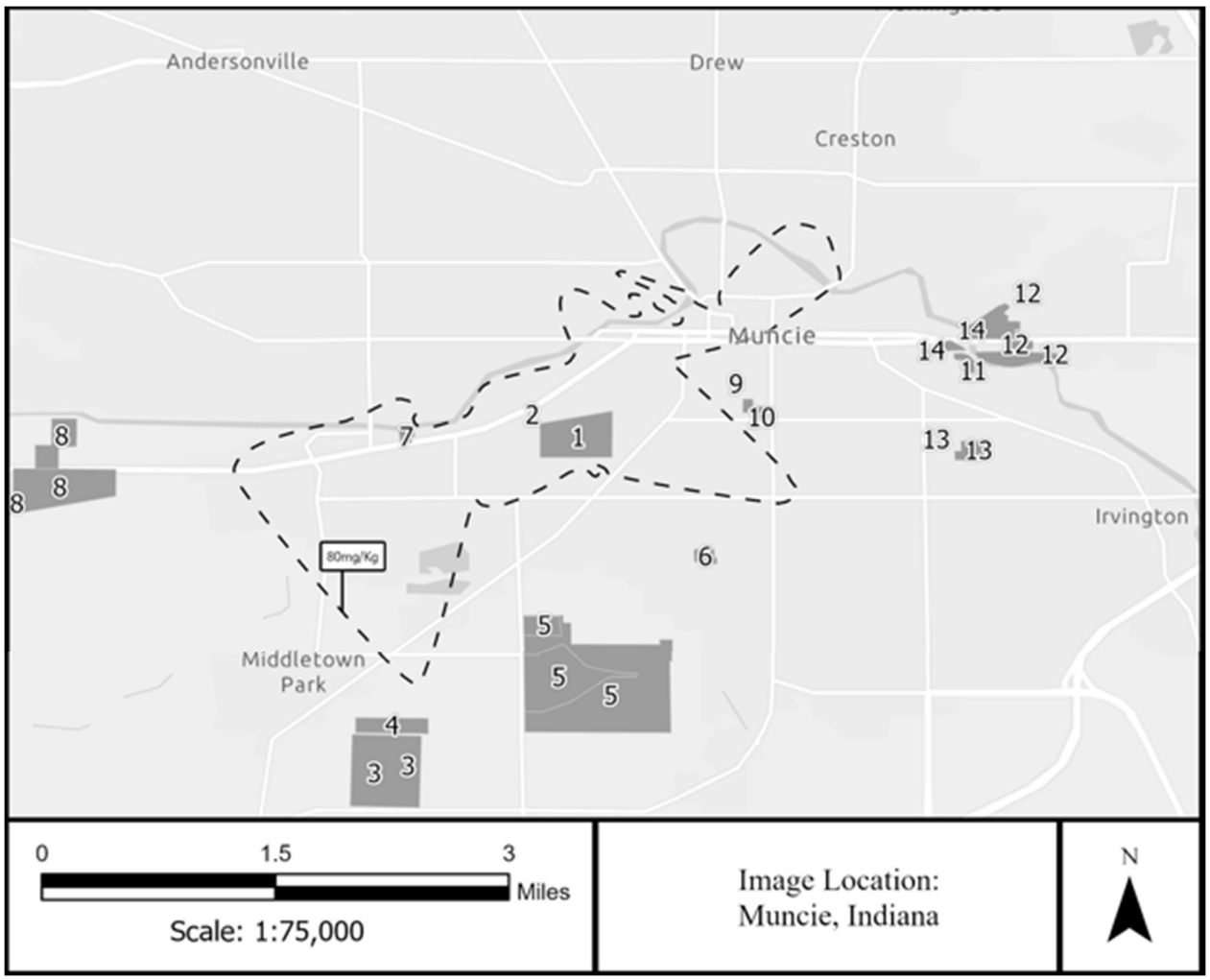

Figure 22. **Current and former Industry:** Refer to Table 5 for in-depth site descriptions

Table 5. Facility information for each of the sites listed in Figure 22. Data were collected from IndianaMAP's map viewer service (IGAW, 2020).

Site Number & Name	Description
1. Manual Transmission of Muncie LLC	Significant Pb contamination on site
2. Custom Chrome and Re-manufacturing Facility	Undesignated metals release noted
3. Delphi Energy & Engine	High levels of Pb contamination at north end of property
4. Exide Battery Disposal Plant	No current soil issues noted; water contamination present
5. Progress Rail Manufacturing Corp.	Significant contamination including Oil, PCBs, Metals including Chromium and Lead
6. WITT Industries Galvanizing Facility	High soil lead contamination
7. West Killgore RCF	Contamination brought on site as fill material – not likely to migrate
8. Borg Warner Plant	Lead contamination reported below levels detected in this study
9. Millennium Place III/IV	Elevated water lead and water chromium levels indicated by Indianamap
10. H&L Plating and Grinding	Soil contamination not identified; Hexavalent chromium in water
11. Manufactured Gas Plant	Significant soil metal contamination – far downwind of iso-zone: worthy of future research
12. Mock's Pond Landfill	Environmental covenant present – capped zinc and lead contaminated soils
13. Frank Foundry	Soil contains lead and chromium – undesignated levels
14. King's Forge	Lead and arsenic contamination – levels undesignated

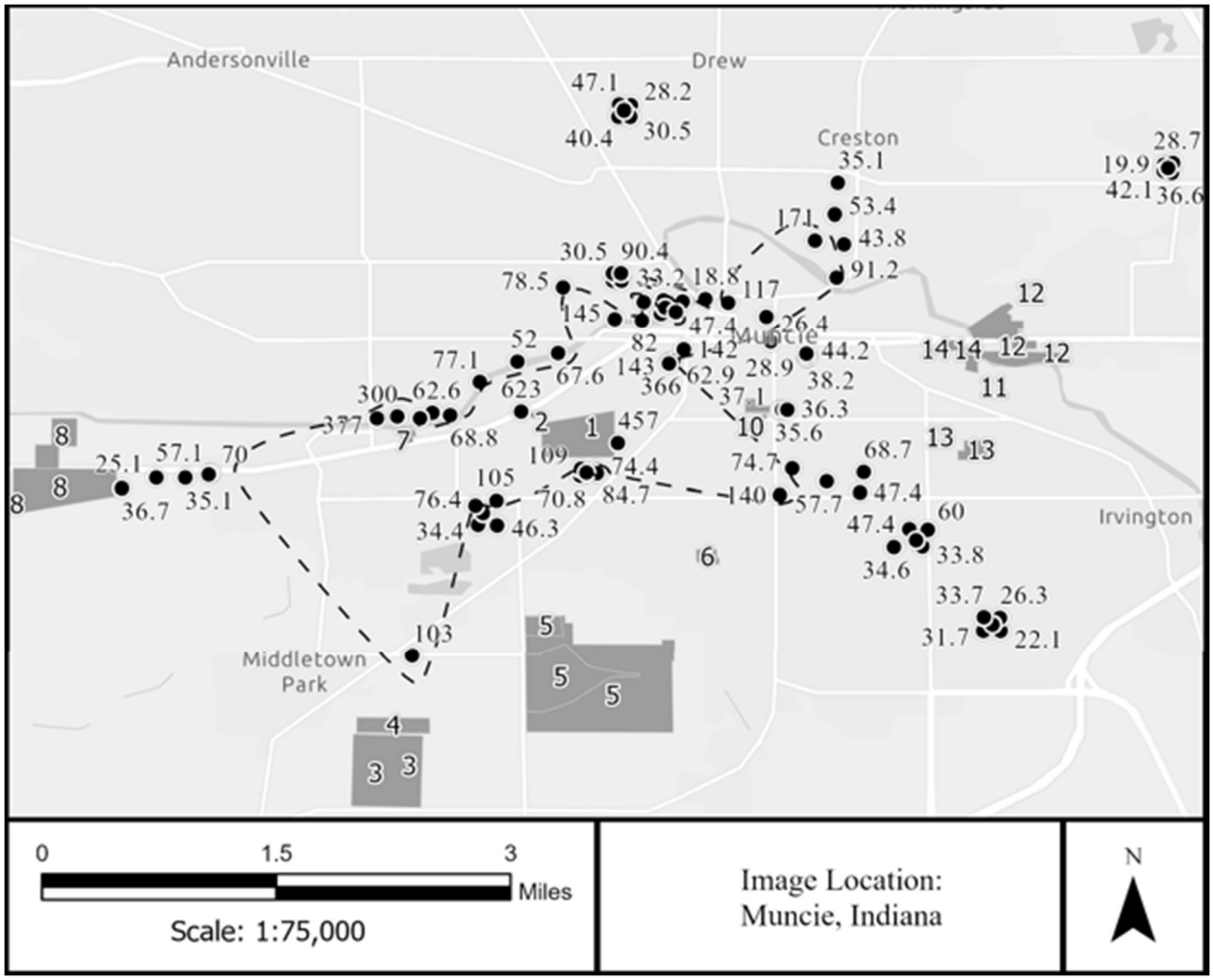

Figure 23. Combined map including both industry and sampled soil-lead levels.

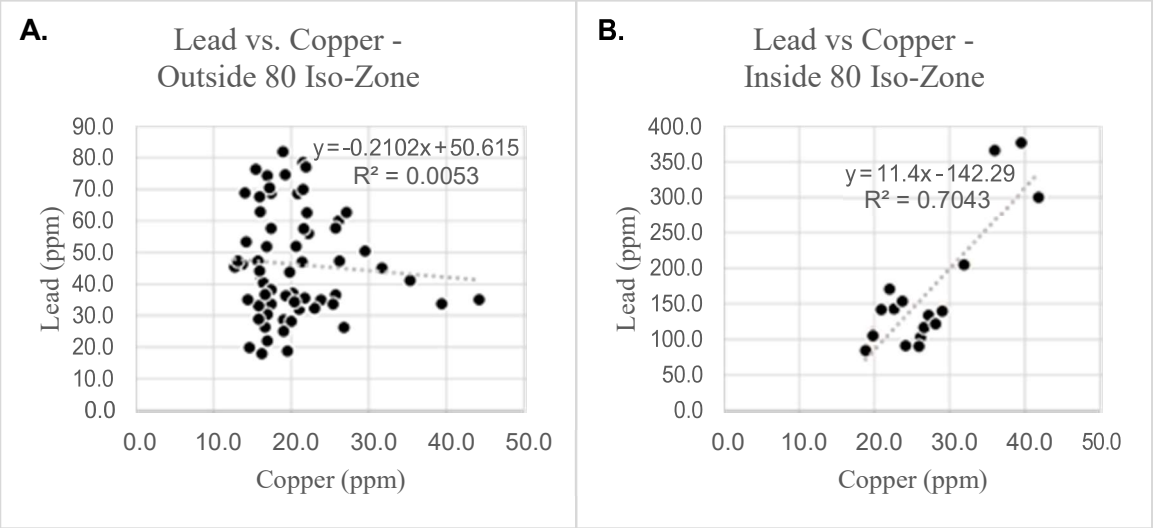

Figure 24. Lead vs copper levels in scatterplots outside (A) and inside (B) the 80 iso-concentration zone. Lead and copper are commonly utilized in battery production as different components (cathode and anode) and thus the interaction between these two metals was explored. Note the Y-axis adjustment on A which shows the lower lead level data.

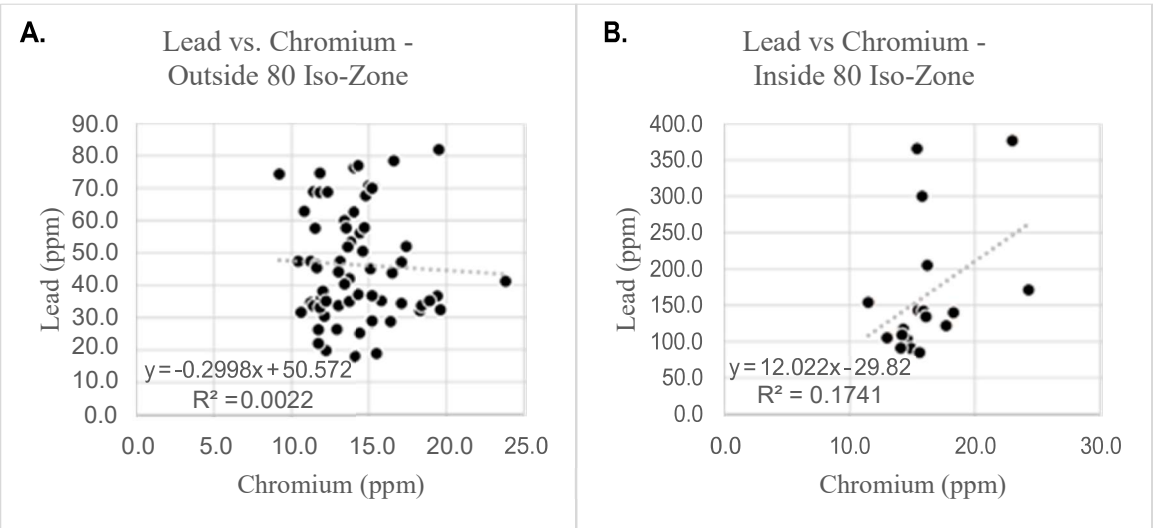

Figure 25. Lead vs chromium levels in scatterplots outside (A) and inside (B) the 80 iso-concentration zone.

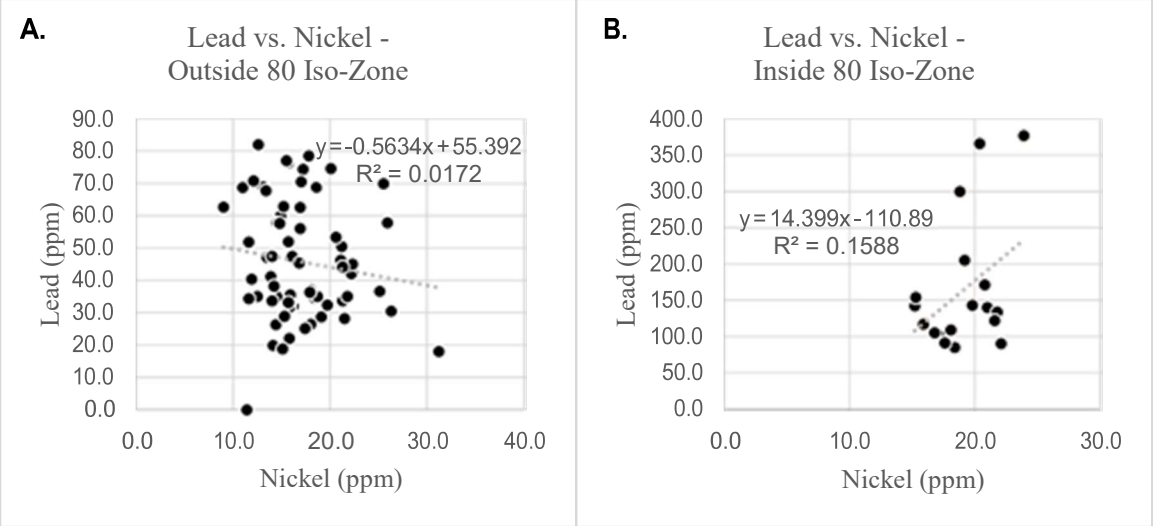

Figure 26. Lead vs nickel levels in scatterplots outside (A) and inside (B) the 80 iso-concentration zone.

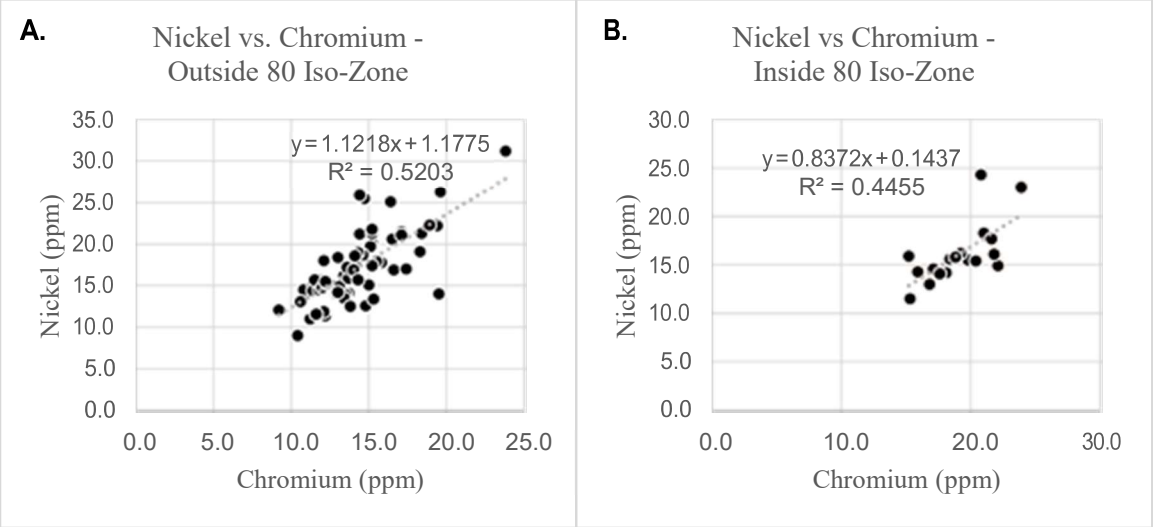

Figure 27. Nickel vs chromium levels in scatterplots outside (A) and inside (B) the 80 iso-concentration zone.

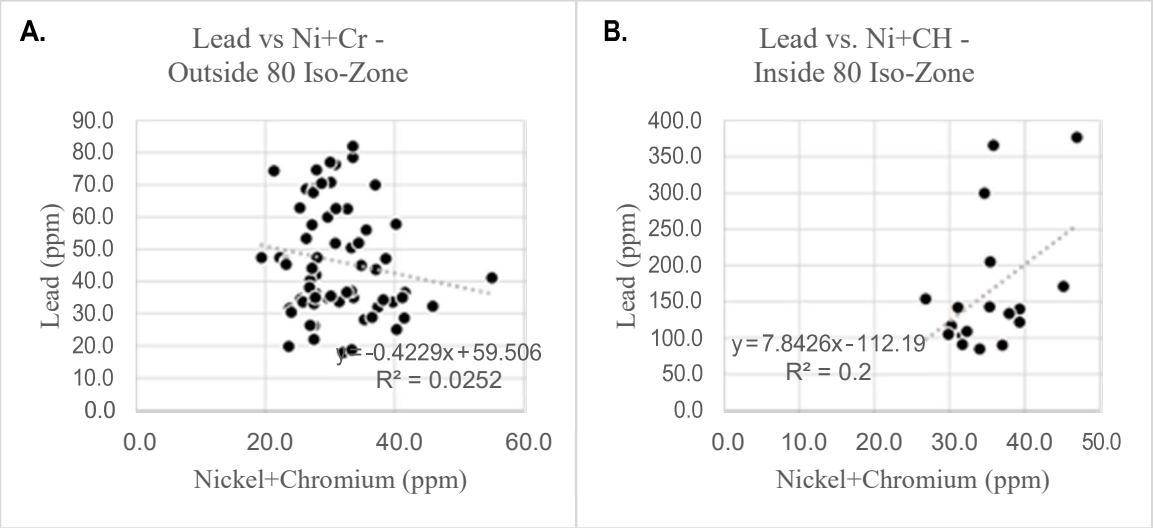

Figure 28. Lead vs the interaction between nickel and chromium levels outside (A) and inside (B) the 80 iso-concentration zone.

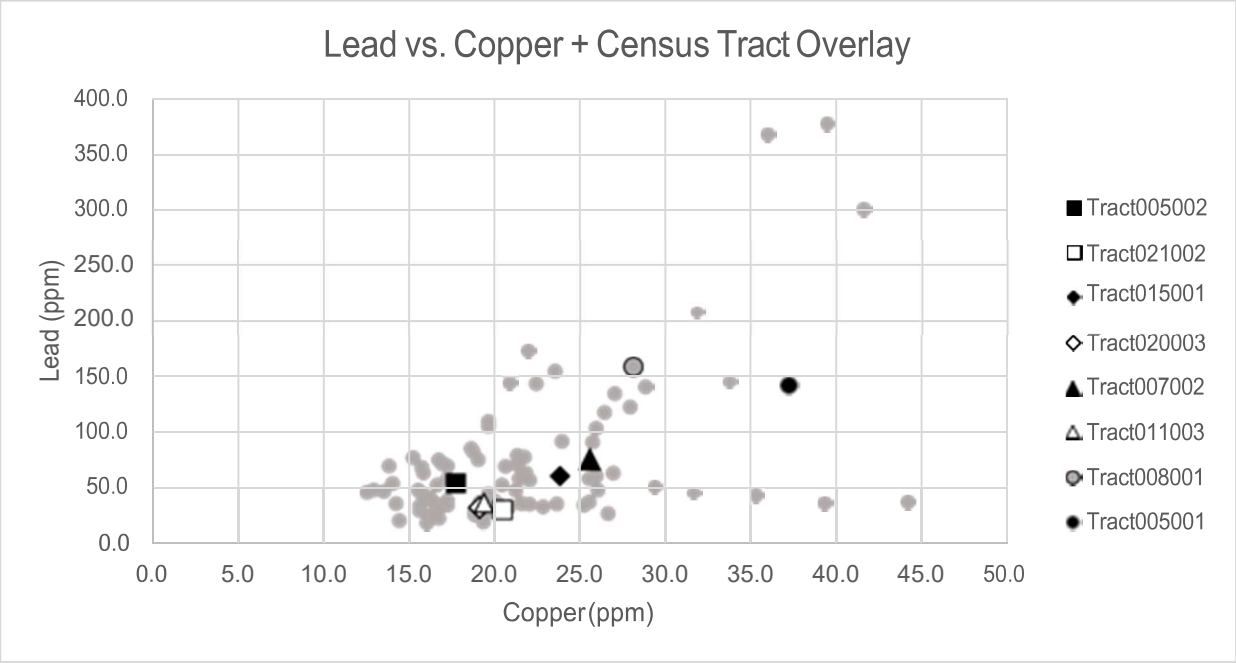

Figure 29. Census tract averages were overlain atop the general data spread from the lead vs. copper graph. A clustering effect is apparent where the majority of sites fall within the spread of the overall dataset. Two tracts fall away from the set; however, they are controlled spatially by proximity to the former battery plant grounds. These tracts are controlled by the high levels of a few points. Refer to Figure 25B for locations of the associated census tract IDs.

Statistics

The study area (the city of Muncie) is 34.6 square miles with a population of 69,233 individuals making the population density average 1997.5 people per square mile (Bureau, 2018). Following the mapping and interpretation of soil samples and associated iso-concentration lines, additional population data were determined from the created polygons. Approximately 7,828 individuals live within the 80 iso-concentration zone, which has an area of 2.77 square miles, making its population density approximately 2826 people per square mile. Given the 146% difference in population density between the 80 iso-concentration zone and overall Muncie area, a correction factor of -1.46 was applied to the number of cases within the 80 iso-concentration zone to fix population density discrepancies. Given the difference in population sizes, normalizing the populations using the aforementioned correction is required to compare the different test result distributions within. Table 6 illustrates the process of normalization.

Approximately 11.3% of the population of Muncie lives within the 80 iso-concentration zone. Of the individuals tested with a BLL result ≥ 1 ppm, a corrected 10.9% reside in the 80 iso-concentration zone. The disparity between percent of cases in the 80 iso-concentration zone versus outside increases when the dataset is limited by test result range. Individuals having a BLL greater than 5 µg/dL in the 80-iso-concentration zone consist of 19.2% of the total cases recorded, greater than 10 µg/dL in the iso-concentration zone are 23.5% of the total cases, and greater than 25 µg/dL in the iso-concentration zone make up 25.2% of the total cases. The adjusted population density of the 80 iso-concentration zone shows a clear increase of frequency of higher level blood Pb contamination with respect to the overall Muncie area (Table 6).

Table 6. Summary table for calculations involving Muncie population corrections.

	Delaware County	Muncie	80 Iso	Density correction factor	Unadjusted %	80 iso pop density correction	% cases pop density correction
Pop	117575	69233	7828		11.3%		
Area (Sq Mi)	397.19	34.66	2.77		8.0%		
Pop Density (Pop/Sq Mi)	296	1997	2826	**1.41**			
Case Total	1177	808	125		15.5%	88.35	10.9%
Cases >5	210	154	42		27.3%	29.69	19.3%
Cases >10	74	57	19		33.3%	13.43	23.6%
Cases >25	18	14	5		35.7%	3.53	25.2%

Linear Regression Analysis

Linear regression statistical analysis explains 6.5% of the variance contributing to the known elevated BLL in the study area (Table 7). The model is statistically significant ($P< .001$, F=10.14) as outlined by Table 8. Input independent variables include the age of a respondent at the time of blood testing, gender, race, season of test, whether or not the respondent resided within the 80 ppm soil iso-concentration zone, percentage of a census tract in poverty, and percentage of a census tract containing houses constructed prior to 1980. Of these initial variables, gender ($P<.01$, t=3.039) and residence within the 80 ppm iso-concentration zone ($P<.001$, t=5.045) are significant. Of the significant results, presence within the 80 iso-concentration zone had the strongest effect followed by gender, while the spring season falls just short of the significance threshold ($P=.056$). Males were slightly more likely to have an elevated BLL, with an average of 4.25 µg/dL against 3.35 µg/dL of females (Table 9). The

remaining initial variables did not have a statistically significant impact on tested residents' blood Pb concentrations.

Table 7.

Linear Regression Analysis Summary			
R	R Square	Adjusted R Square (%)	Std. Error of the Estimate
0.269	0.073	**6.5**	5.079

Table 8.

ANOVA					
	Sum of Squares	df	Mean Squares	F	Sig.
Regression	2355.498	9	261.722	10.144	**0.000**
Residual	30108.518	1167	25.800		
Total	32464.015	1176			

Table 9. Summary of average BLLs for each gender.

Gender	Male	Female
Individuals tested	622	555
Avg. BLL by gender (µg/dL)	4.25±6.24	3.35±3.80

One-to-one interactions for all variables were analyzed in relation to BLL. Of the 52 interactions, only four were found to have statistical significance ($P<.05$). The interaction between age at the time of test and the gender variable ($P<.001$, t=5.603) displayed the strongest contribution to increased blood Pb concentration of all variables. Remaining interacting variables contribute relatively equally to BLL. The interaction between the age at the time of

test and the spring season has the next strongest impact on BLL ($P<.01$, t= 3.118). The interaction between the 80 soil iso-concentration zone and gender ($P<.05$ t=2.509) as well as between the 80 soil iso-concentration zone and the summer season ($P<.05$, t=2.210) displayed the next highest contribution to elevated BLL and are statistically significant ($P<.05$). A summary of variables and interacting variables is included as Table 10.

Table 10. Coefficients shows significance of each individual variable contributing to BLL as well as contributing interactions between variables. Standardized coefficients Beta shows the relative strength of each variable in relation to the other variables being analyzed. Collinearity tolerance and Statistics VIF explain likelihood of variables affecting one another (values closer to 1 = least likely to interact).

Coefficients							
	B*	Coefficients Std. Error	Beta**	t	Sig (*P*)	Collinearity Tolerance	Statistics VIF
(Constant)	2.888	0.275		10.495	0.000		
Int. Between Age at Test and Gender	0.082	0.015	0.158	5.603	**0.000**	0.995	1.005
Soil_IsoZone80	2.433	0.482	0.143	5.045	**0.000**	0.993	1.007
Int. between Age at Test and Spring	0.051	0.016	0.088	3.118	**0.002**	0.988	1.013
Gender	0.904	0.297	0.086	3.039	**0.002**	0.994	1.006
Int. Between Soil IsoZone80 and Gender	2.426	0.967	0.071	2.509	**0.012**	0.993	1.007
Int. Between Soil IsoZone80 and Summer	2.544	1.151	0.063	2.210	**0.027**	0.988	1.012
Spring Test	0.705	0.368	0.057	1.916	0.056	0.885	1.130
Summer Test	0.082	0.362	0.007	0.226	0.821	0.993	1.132
Age at Time of Test	0.001	0.007	0.003	0.109	0.913	0.992	1.008

* "B" represents the unstandardized B coefficient
** "Beta" represents the Standardized Coefficients Beta

Discussion

Elevated Blood and Soil Pb Levels

Elevated BLLs plotted within the census tracts of Muncie display distribution traits similar to other metropolitan areas such as Indianapolis, Indiana (Filippelli et al., 2005; Morrison et al., 2013). The distribution of elevated BLLs is most concentrated toward the Muncie city center (Figures 6 through 9), excluding large factory footprints (Morrison et al., 2013). The BLL dataset provided by Regenstrief Institute ranges from less than 1 µg/dL to a maximum of 55 µg/dL and displays a similar pattern of blood concentrations increasing with proximity to the city center, as outlined by Morrison (2013).

Soil samples collected from fall of 2018 through summer of 2019 had bio-accessible soil-lead concentrations peak in the city center and decrease toward less populace areas, as shown in Figures 21 and 23. This matches the bio-accessible soil Pb patterns of the Filippelli et al. (2005) study in Indianapolis. The peak zone of bio-accessible soil-lead at 628 ppm and 457 ppm from two points encompasses the northern portion of the former Manual Transmission of Muncie facility in Figure 26. Samples collected by the RACER Trust at the former Delco Remy battery factory range between 14,000 ppm and 40,000 ppm bio-accessible Pb (EPA method 3050b) (CRA, 2011). Sample collection for this study was limited to areas of public right-of-way, and access to the former battery facility grounds was not possible to obtain confirmatory samples.

There is clear disparity between the Ginn woods average background bio-accessible soil-lead of 29.6±16.4 ppm versus the 84.01±96.82 ppm found primarily in Muncie census tracts. The background levels were measured from Ginn Woods in this project and compared to those taken statewide by the USGS (EPA, 2018). However, the difference between the measured bio-accessible 29.6±16.4 ppm background soil-lead level in Ginn woods and the USGS measured state wide average of 30.7±54.4 ppm total Pb is negligible (EPA, 2018). The 80 iso-

concentration zone is the primary contributor to the trend in elevated soil Pb concentrations when compared to other variables, and it is a major zone of interest for this study. Through statistical analysis, out of the significant variables, 3 of 7 (Soil_IsoZone80, Int. Between Soil IsoZone80 and Gender, Int. Between Soil IsoZone80 and summer) were directly related to the 80 iso-concentration zone, with the second strongest influence in the model belonging to the 80 iso-concentration zone alone. See Table 10 for a full list of variables.

Significant Variables

The population density corrected dataset outlined in Table 6 tests for irregular distributions of test results. Comparing total individuals within the 80 iso-concentration zone and the results of those tested yielded 11.3% and 10.9% of the population respectively. The expectation, then, is for the test brackets (≥ 5 µg/dL, ≥ 10 µg/dL, ≥ 25 µg/dL) to reflect this similarity. Using the earlier population density correction, smaller groups of BLL were identified inside the 80 iso-concentration zone with concentrations of 5, 10, and 25 µg/dL. The percentage of cases in each of the identified groups fell to 19.2%, 23.5%, and 25.2% respectively. The percent of cases within these sub-groups was higher than the expected 10.9% to 11.3% of the population. Thus, the assumption that the tested population (within the 80 iso- concentration zone) would reflect the total population (overall) failed as per Table 6. The correspondence between higher numbers of elevated BLL test results and the 80 iso- concentration zone is most likely from anthropogenic interaction. Overall, when all blood Pb reports are factored in, Muncie has an expected ratio of tested population versus total population with respect to the 80 iso-concentration zone at 10.9% and 11.3% respectively. At higher blood Pb results, the tested individuals become more concentrated within the area of the 80 iso-concentration zone. Considering the information from Table 6, the statistical analysis supports the claim that living within the 80 iso-concentration zone contributes considerably to blood Pb concentrations when

compared to the other significant variables. The strongest variable, age at time of test, is naturally biased due to the number of individuals tested at younger ages (Table 10). Location inside or outside the 80 iso-concentration zone, whether as a stand-alone variable or as an interacting variable, is shown to influence an individual's BLL. Being exposed to conditions with elevated soil-lead levels leads to elevated BLL with sufficient soil contact due to the many potential routes of exposure. Inhalation of particulate, breathing rate, gastrointestinal bio-availability, rate of ingestion constants, average soil ingested per day, area of skin exposure, and days per week of exposure all factor into determining an individual's BLL (DTSC, 2011).

Of the interacting significant variables, age at time of test and the spring season (P=.002) are the least quantifiably explainable. No apparent links between the two variables were found at the time of analysis.

Use of outdoor spaces with regard to children's play varies considerably with each season (Naylor, 1985). The near-significance of spring months (P=.056) can be explained by longer daylight hours as well as favorable weather conditions (Naylor, 1985). When taking the 80 iso-concentration zone into consideration, summer (P=.027) months are significant, which is expected due to the longer more comfortable days in combination with children having greater access to the outdoors. This allows for greater contact with Pb containing soils within the 80 iso-concentration zone.

The positive effect of gender on blood Pb contamination levels can be attributed to multiple potential causes and must be split into separate age categories. Children under the age of 5 typically are required to stay in close proximity to their homes (Naylor, 1985). Children of this age tend to find small patches of bare ground that is more interesting than the grass covered areas of a yard (Hart, 1979). Differences in gender of young (5 or younger) children and play in the dirt is not something observed to naturally occur (Wood, 1993). If there is an apparent difference in

play, it is more than likely attributed to a parent's influence, pushing a young child toward one activity over another rather than historic stereotypes (Pomerleau et al., 1990).

For children between the ages of 6 and 12, there is a trend of boys traveling farther from home and preferring wild areas to predefined play areas such as parks, whereas girls have a typically more restricted wander range (Naylor, 1985). Regardless of social status, it is noted that children of urban environments, which downtown Muncie is considered, favor unofficial zones for play such as the streets, illegal waste dumps, and streams. (Naylor, 1985). The combination of boys' larger travel range from home and the propensity for exploration of unplanned zones for play could easily lead to higher likelihood of Pb exposure in the 80 iso- concentration zone.

Individuals of working age fall into a different category, and with Muncie's manufacturing history, factory work, specifically in the various former and current industrial complexes, could be another explanation for increased BLLs. Gender differences in blue collar work vary wildly for different positions: Craftsmen are 91% and laborers are 78% male respectively while administrative positions are 79% female (Gabriel and Schmitz, 2007). Craftsmen and laborers are much more likely to come into contact with contaminants of any sort in factory work when compared to the office work.

Notable Insignificant Variables

An interesting association was identified, wherein the summer season alone has no significance (P=.821) on the model, but its interaction with the 80 iso-concentration zone is significant (P=.027). Even though it is possible to assume that the 80 iso-concentration zone is strong enough to influence other variables, it is not the case when the 80 iso-concentration zone was interacted with every other variable in the model. This interaction experiment resulted in only two significant results of 12 possible interactions.

There are not enough data on different ethnicities to provide useful data comparisons. The lack of significance for any of the ethnicities and BLL as well as the interaction between age, gender, the 80-iso concentration zone, or season can be attributed to the significantly smaller sample sizes compared to the Caucasian group.

The respondents' age at time of blood Pb testing was not significant (P=.913) which is not surprising due to the high number of tests done on very young children (1-2 years old). If a wider range of BLL samples were taken from a more evenly distributed age range, potential significance could become apparent.

No interacting variables for housing age or poverty status produced a result that approached significant levels (P<.05). The lack of significance of any value related to housing age or poverty rates was surprising, which suggests that regardless of the age of a home or poverty status, Pb can be a problem. It is clear that further refinement of soil surveying is required to confirm this statistical anomaly. One would expect a relation to form between housing age and BLL, due to the presence of lead-based paints.

While significant (P<.001), this model and all contributing factors including residing in the 80 iso-concentration zone are only weakly correlative to blood Pb concentrations. See

Tables 7 through 10. The model explains 6.5% of the factors that go into determining an individual's BLL. The low result is not unexpected due to the variety of routes of Pb contamination with which an individual may come in contact. Potential sources of Pb contamination range from lead-based paint in houses constructed prior to 1978, water supply pipes in systems containing older infrastructure pre-dating 1986, dust, contaminated soils, herbal medications or toys from foreign countries where Pb use is not banned, and in many occupations and hobbies (CDC, 2019; Congress, 1986). While Pb exposure is a more acute danger to the hobbyist or worker, an individual may inadvertently carry Pb dust on their clothes back to their residence where family or friends may be affected (CDC, 2019). Careers involving Pb include but are not limited to abatement or cleanup of contaminated facilities, structural demolition, artisanal decorating – such as leaded glazing of ceramics, mining, and working or using firearms (CDC, 2019). Hobbies that can involve Pb in a similar way are related to the careers, such as working with older vehicles, primarily involving welding or soldering. Further, an individual may contact Pb at a location far from their home, adding potential for error. An explanation of variance above that of this study was achieved by Bradham (2017) in their study comparing total Pb and bio-accessible Pb extracted from the soils of a child's play area, just outside their home. The study found 25% and 28% explanation of variance for total and bio- accessible Pb respectively. Thus, the 6.5% of variance explained in this study of potentially distal parks and roadsides can still be considered an important contributing factor of blood Pb contamination.

Additional Soil Analytes

Soil samples collected in this study were analyzed for bio-accessible cadmium, chromium, copper, and nickel in addition to Pb. Cadmium exposure can cause significant health problems ranging from heart and lung damage to bone and tooth degradation (CDC, 1976). The presence of Cd above five ppm was not detected in any of the collected samples (Table 3). Of the

three other metals, chromium has the highest potential to cause harm with exposure. Chromium exists in two oxidation states, Cr(III) and Cr(VI). Cr(III), in the correct dosages, is healthy while Cr(VI) or hexavalent chromium exposure can cause serious adverse health effects such as nasal damage, stomach ulcers, dizziness, weakness, or cancer (Guertin, 2005). Copper and nickel by comparison have significantly more limited health impacts. These metals are all fairly common in the manufacturing of batteries and other industrial processes common to the Muncie area.

Figures 12 through 29 outline relations between the various metals involved in industrial processing sampled by this study. Several points, notably the two points located immediately northwest and southeast of the former Manual Transmission of Muncie facility were excluded from the dataset due to their status as extreme outliers based on statistics. These points, when included, influenced the graphs immensely. In some cases, they completely controlled the entire dataset. Upon their removal, the graphs could be analyzed more accurately. The above-mentioned NW and SE points are excluded in all discussed graphs. In Figure 12 overall lead vs. copper and overall lead vs. chromium, both A) and B) have R^2 values of 0.29 and 0.12, indicating weak and very weak correlation respectively.

Figures 13 through 15 display limited to no correlation between contaminant levels, north vs. south of the White River for lead vs. copper, lead vs. chromium, and lead vs. nickel. An additional analysis was undertaken shown in Figure 16, outlining nickel vs. chromium north vs. south of the White River. The R^2 values for nickel vs. chromium north vs south of the White River are 0.021 and 0.48, respectively. Nickel and chromium are commonly utilized in Ni-chrome alloys; however, it is unknown whether this alloy was used in any Muncie manufacturing industries.

Average contaminants within Muncie census tracts were graphed in Figure 17. It was clear that no correlations for metals were apparent with the exception of lead vs. copper. This could be explained spatially by clustering effects to potentially identify other chemical sources of the metals. Figures 18 and 19 were graphed and mapped to help determine whether sample locations within clusters identified in earlier graphs contained spatial relationships. Inter-sample site variation was apparent and at times large, as indicated in Figures 18 and 19 with Clifton Wallace Park. Clusters of sample groups contained points from parks located on the opposite side of the city, while some parks had points across two distinct groups within the graph.

The lead vs. copper correlation mentioned above contributes to the potential source of contamination pointing to the former Delco Remy battery plant. Further mapping and graphing were required to confirm this potential source spatially. Three distinct clusters in census tract averages for lead vs. copper are outlined in Figure 20. These are separated into low, mid-range, and high lead-copper clusters. The associated map shows one distinct group as spatially significant. The highest lead vs. copper points are located closest to the former Delco Remy battery plant. The low and mid-range clusters contain points which are not spatially related, instead spread throughout the Muncie tracts. Potential for brownfield or older industry to be causing increased soil concentrations is unlikely due to the lack of metal contaminated brownfields in every census tract identified.

Figure 24, A) lead vs. copper outside the 80 iso-zone, and B) lead vs. copper inside the 80 iso-zone display a different story entirely. Figure 24 A) contains an R^2 value of 0.05 while B) has an R^2 value of 0.70. This difference between lead and copper versus iso-concentration zone should be considered significant. Referencing Figure 21 through 23, and Table 5 shows that the levels within the 80 iso-concentration zone could *primarily* be contributed to the Manual Transmission of Muncie property (former Delco Remy battery manufacturing plant), and/or

Custom Chrome and Re-manufacturing facility. The proximity to the former Delco Remy battery plant is a potential cause for the lead and copper association, given the prevalent use of both metals in the manufacturing process of batteries. *Secondary-potential* sources were identified as the former Progress Rail Manufacturing Corporation and WITT Industries Galvanizing Facility. The secondary sources are not located within the 80 iso-concentration zone; however, they are potentially upwind of the zone.

Figure 25 compares lead vs. chromium outside and inside the 80 iso-concentration zone with similar but weaker R^2 trends to those identified in Figure 24 with values of 0.002 and 0.17. These values are not indicative of individual trends within the data. Figure 26 examines lead vs nickel outside vs. inside the concentration zone; however, the R^2 values of 0.01 and 0.15 show limited effect, if any, of the presence of the iso zone. Figure 27 identifies potential correlation between nickel and chromium levels both inside and outside the iso-concentration zone. The widespread industrialization within the Muncie area may have contributed to the correlation identified in Figure 27. There are various locations around the city with contamination of chromium in soil and water; however, the extent of contamination is not known as further mapping is needed.

Figure 29 relates lead vs. copper with overlain census tract averages. This graph shows the majority of census tracts falling within the 'normal' distribution of the copper vs. lead graph. Two census tracts, located adjacent to or containing the former battery plant, are outliers from the normally distributed group. These contain the highest lead vs copper averages. The proximity to the battery facility is then one of the strongest, if not the strongest indicator for soil contamination of lead and copper.

A stand-out point known as the "DIY point" requires additional attention that this study was unable to address. The point is located south west of the Delco Remy battery plant's

location, upwind of the facility, but downwind of both the Exide structure and former Delphi Energy & Engine. This point requires significant attention, in that the study area could have two plumes of soil-lead contamination. It is the south-western most in the sample array, and its proximity to the above mentioned two facilities could contribute to a different contamination issue. For the purposes of this study, however, the point is included in the initial 80 iso-concentration zone. Future work should seek to perform additional sampling up and downwind of this point.

Conclusions

Bio-accessible soil Pb levels within the city of Muncie census tracts exhibit more than double the average background total soil-lead concentrations, primarily due to the 80 iso-concentration zone.

The similarities between the corrected population of tested individuals and overall population of Muncie display an increase in blood Pb cases at 5, 10, and 25 µg/dL within the 80 soil iso-concentration zone. The cases in these groups depart from the ratio of the general population living inside vs. outside the 80 soil iso-concentration zone both at the ≥ 1 µg/dL BLL and in terms of general inhabitants.

Statistical analysis indicates a weak correlation between analyzed variables and blood Pb concentrations ($P<.001$).

Co-lateral investigations conducted by the RACER Trust and this study found elevated soil-lead levels in the residential zones surrounding the former Manual Transmission of Muncie property. These independent studies offer confirmatory analytical results.

The former Delco Remy battery plant is likely the primary contributor to the elevated soil-lead levels surrounding the former Manual Transmission of Muncie facility property. The findings of soil-lead concentrations beneath the former battery plant of 40,000 ppm - more than 1300 times the measured background soil-lead level of 29.6±16.4 ppm and a deposit of 570,000 ppm total Pb on the facility's roof reinforces this assertion.

Future Work

Continued research into contamination routes is necessary for refinement of the statistical model, such as groundwater sampling, and additional soil sampling to further delineate the iso-concentration lines in order to afford protection to the citizens living in Muncie. The "DIY Point" in the south western portion of Muncie provides a future springboard for continued research. Its location just north of two battery related facilities, the Excide plant, and the former Delphi Energy & Engine, plant is cause for concern, with a potential secondary lead concentration zone overlapping the first. Further sampling both upwind and downwind of these battery facilities is important in designating problem or hotspots for soil-lead. Additional sampling is necessary on site and surrounding the former Manual Transmission plant and Delco Remy facility grounds. This sampling will allow further delineation of high soil-lead levels with the potential to impact Muncie children. Further, a targeted bio-accessibility assay performed similar to the Philadelphia study conducted by Bradham et al, (2017) could offer significantly improved statistical accuracy when determining blood and soil lead interaction. The methodology would require contacting individuals and accessing specific properties; however, this increased level of intrusion would certainly be fruitful in increased scientific accuracy.

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Appendix A

EPA Method 3050B

METHOD 3050B

ACID DIGESTION OF SEDIMENTS, SLUDGES, AND SOILS

1.0 SCOPE AND APPLICATION

1.1 This method has been written to provide two separate digestion procedures, one for the preparation of sediments, sludges, and soil samples for analysis by flame atomic absorption spectrometry (FLAA) or inductively coupled plasma atomic emission spectrometry (ICP-AES) and one for the preparation of sediments, sludges, and soil samples for analysis of samples by Graphite Furnace AA (GFAA) or inductively coupled plasma mass spectrometry (ICP-MS). The extracts from these two procedures are not interchangeable and should only be used with the analytical determinations outlined in this section. Samples prepared by this method may be analyzed by ICP-AES or GFAA for all the listed metals as long as the detecion limits are adequate for the required end-use of the data. Alternative determinative techniques may be used if they are scientifically valid and the QC criteria of the method, including those dealing with interferences, can be achieved. Other elements and matrices may be analyzed by this method if performance is demonstrated for the analytes of interest, in the matrices of interest, at the concentration levels of interest (See Section 8.0). The recommended determinative techniques for each element are listed below:

FLAA/ICP-AES		GFAA/ICP-MS
Aluminum	Magnesium	Arsenic
Antimony	Manganese	Beryllium
Barium	Molybdenum	Cadmium
Beryllium	Nickel	Chromium
Cadmium	Potassium	Cobalt
Calcium	Silver	Iron
Chromium	Sodium	Lead
Cobalt	Thallium	Molybdenum
Copper	Vanadium	Selenium
Iron	Zinc	Thallium
Lead		
Vanadium		

1.2 This method is not a total digestion technique for most samples. It is a very strong acid digestion that will dissolve almost all elements that could become "environmentally available." By design, elements bound in silicate structures are not normally dissolved by this procedure as they are not usually mobile in the environment. If absolute total digestion is required use Method 3052.

2.0 SUMMARY OF METHOD

2.1 For the digestion of samples, a representative 1-2 gram (wet weight) or 1 gram (dry weight) sample is digested with repeated additions of nitric acid (HNO_3) and hydrogen peroxide (H_2O_2).

2.2 For GFAA or ICP-MS analysis, the resultant digestate is reduced in volume while heating and then diluted to a final volume of 100 mL.

2.3 For ICP-AES or FLAA analyses, hydrochloric acid (HCl) is added to the initial digestate and the sample is refluxed. In an optional step to increase the solubility of some metals (see Section 7.3.1: NOTE), this digestate is filtered and the filter paper and residues are rinsed, first

with hot HCl and then hot reagent water. Filter paper and residue are returned to the digestion flask, refluxed with additional HCl and then filtered again. The digestate is then diluted to a final volume of 100 mL.

2.4 If required, a separate sample aliquot shall be dried for a total percent solids determination.

3.0 INTERFERENCES

3.1 Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed in accordance with the quality control requirements given in Sec. 8.0 to aid in determining whether Method 3050B is applicable to a given waste.

4.0 APPARATUS AND MATERIALS

4.1 Digestion Vessels - 250-mL.

4.2 Vapor recovery device (e.g., ribbed watch glasses, appropriate refluxing device, appropriate solvent handling system).

4.3 Drying ovens - able to maintain 30°C ± 4°C.

4.4 Temperature measurement device capable of measuring to at least 125°C with suitable precision and accuracy (e.g., thermometer, IR sensor, thermocouple, thermister, etc.)

4.5 Filter paper - Whatman No. 41 or equivalent.

4.6 Centrifuge and centrifuge tubes.

4.7 Analytical balance - capable of accurate weighings to 0.01 g.

4.8 Heating source - Adjustable and able to maintain a temperature of 90-95°C. (e.g., hot plate, block digestor, microwave, etc.)

4.9 Funnel or equivalent.

4.10 Graduated cylinder or equivalent volume measuring device.

4.11 Volumetric Flasks - 100-mL.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is questionable, analyze the reagent to determine the level of impurities. The reagent blank must be less than the MDL in order to be used.

5.2 Reagent Water. Reagent water will be interference free. All references to water in the method refer to reagent water unless otherwise specified. Refer to Chapter One for a definition of reagent water.

5.3 Nitric acid (concentrated), HNO_3. Acid should be analyzed to determine level of impurities. If method blank is < MDL, the acid can be used.

5.4 Hydrochloric acid (concentrated), HCl. Acid should be analyzed to determine level of impurities. If method blank is < MDL, the acid can be used.

5.5 Hydrogen peroxide (30%), H_2O_2. Oxidant should be analyzed to determine level of impurities. If method blank is < MDL, the peroxide can be used.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

6.2 All sample containers must be demonstrated to be free of contamination at or below the reporting limit. Plastic and glass containers are both suitable. See Chapter Three, Section 3.1.3, for further information.

6.3 Nonaqueous samples should be refrigerated upon receipt and analyzed as soon as possible.

6.4 It can be difficult to obtain a representative sample with wet or damp materials. Wet samples may be dried, crushed, and ground to reduce subsample variability as long as drying does not affect the extraction of the analytes of interest in the sample.

7.0 PROCEDURE

7.1 Mix the sample thoroughly to achieve homogeneity and sieve, if appropriate and necessary, using a USS #10 sieve. All equipment used for homogenization should be cleaned according to the guidance in Sec. 6.0 to minimize the potential of cross-contamination. For each digestion procedure, weigh to the nearest 0.01 g and transfer a 1-2 g sample (wet weight) or 1 g sample (dry weight) to a digestion vessel. For samples with high liquid content, a larger sample size may be used as long as digestion is completed.

> NOTE: All steps requiring the use of acids should be conducted under a fume hood by properly trained personnel using appropriate laboratory safety equipment. The use of an acid vapor scrubber system for waste minimization is encouraged.

7.2 For the digestion of samples for analysis by GFAA or ICP-MS, add 10 mL of 1:1 HNO_3, mix the slurry, and cover with a watch glass or vapor recovery device. Heat the sample to 95°C ± 5°C and reflux for 10 to 15 minutes without boiling. Allow the sample to cool, add 5 mL of concentrated HNO_3, replace the cover, and reflux for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3, repeat this step (addition of 5 mL of conc. HNO_3) over and over until no brown fumes are given off by the sample indicating the complete reaction with HNO_3. Using a ribbed watch glass or vapor recovery system, either allow the solution to evaporate to approximately 5 mL without boiling or heat at 95°C ± 5°C without boiling for two hours. Maintain a covering of solution over the bottom of the vessel at all times.

NOTE: Alternatively, for direct energy coupling devices, such as a microwave, digest samples for analysis by GFAA or ICP-MS by adding 10 mL of 1:1 HNO_3, mixing the slurry and then covering with a vapor recovery device. Heat the sample to 95°C ± 5°C and reflux for 5 minutes at 95°C ± 5°C without boiling. Allow the sample to cool for 5 minutes, add 5 mL of concentrated HNO_3, heat the sample to 95°C ± 5°C and reflux for 5 minutes at 95°C ± 5°C. If brown fumes are generated, indicating oxidation of the sample by HNO_3, repeat this step (addition of 5 mL concentrated HNO_3) until no brown fumes are given off by the sample indicating the complete reaction with HNO_3. Using a vapor recovery system, heat the sample to 95°C ± 5°C and reflux for 10 minutes at 95°C ± 5°C without boiling.

7.2.1 After the step in Section 7.2 has been completed and the sample has cooled, add 2 mL of water and 3 mL of 30% H_2O_2. Cover the vessel with a watch glass or vapor recovery device and return the covered vessel to the heat source for warming and to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the vessel.

NOTE: Alternatively, for direct energy coupled devices: After the Sec. 7.2 "NOTE" step has been completed and the sample has cooled for 5 minutes, add slowly 10 mL of 30% H_2O_2. Care must be taken to ensure that losses do not occur due to excessive vigorous effervesence. Go to Section 7.2.3.

7.2.2 Continue to add 30% H_2O_2 in 1-mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.

NOTE: Do not add more than a total of 10 mL 30% H_2O_2.

7.2.3 Cover the sample with a ribbed watch glass or vapor recovery device and continue heating the acid-peroxide digestate until the volume has been reduced to approximately 5 mL or heat at 95°C ± 5°C without boiling for two hours. Maintain a covering of solution over the bottom of the vessel at all times.

NOTE: Alternatively, for direct energy coupled devices: Heat the acid-peroxide digestate to 95°C ± 5°C in 6 minutes and remain at 95°C ± 5°C without boiling for 10 minutes.

7.2.4 After cooling, dilute to 100 mL with water. Particulates in the digestate should then be removed by filtration, by centrifugation, or by allowing the sample to settle. The sample is now ready for analysis by GFAA or ICP-MS.

7.2.4.1 Filtration - Filter through Whatman No. 41 filter paper (or equivalent).

7.2.4.2 Centrifugation - Centrifugation at 2,000-3,000 rpm for 10 minutes is usually sufficient to clear the supernatant.

7.2.4.3 The diluted digestate solution contains approximately 5% (v/v) HNO_3. For analysis, withdraw aliquots of appropriate volume and add any required reagent or matrix modifier.

7.3 For the analysis of samples for FLAA or ICP-AES, add 10 mL conc. HCl to the sample digest from 7.2.3 and cover with a watch glass or vapor recovery device. Place the sample on/in the heating source and reflux at 95°C ± 5°C for 15 minutes.

NOTE: Alternatively, for direct energy coupling devices, such as a microwave, digest samples for analysis by FLAA and ICP-AES by adding 5 mL HCl and 10 mL H_2O to the sample digest from 7.2.3 and heat the sample to 95°C ± 5°C, Reflux at 95°C ± 5°C without boiling for 5 minutes.

7.4 Filter the digestate through Whatman No. 41 filter paper (or equivalent) and collect filtrate in a 100-mL volumetric flask. Make to volume and analyze by FLAA or ICP-AES.

NOTE: Section 7.5 may be used to improve the solubilities and recoveries of antimony, barium, lead, and silver when necessary. These steps are optional and are not required on a routine basis.

7.5 Add 2.5 mL conc. HNO_3 and 10 mL conc. HCl to a 1-2 g sample (wet weight) or 1 g sample (dry weight) and cover with a watchglass or vapor recovery device. Place the sample on/in the heating source and reflux for 15 minutes.

7.5.1 Filter the digestate through Whatman No. 41 filter paper (or equivalent) and collect filtrate in a 100-mL volumetric flask. Wash the filter paper, while still in the funnel, with no more than 5 mL of hot (~95°C) HCl, then with 20 mL of hot (~95°C) reagent water. Collect washings in the same 100-mL volumetric flask.

7.5.2 Remove the filter and residue from the funnel, and place them back in the vessel. Add 5 mL of conc. HCl, place the vessel back on the heating source, and heat at 95°C ± 5°C until the filter paper dissolves. Remove the vessel from the heating source and wash the cover and sides with reagent water. Filter the residue and collect the filtrate in the same 100-mL volumetric flask. Allow filtrate to cool, then dilute to volume.

NOTE: High concentrations of metal salts with temperature-sensitive solubilities can result in the formation of precipitates upon cooling of primary and/or secondary filtrates. If precipitation occurs in the flask upon cooling, do not dilute to volume.

7.5.3 If a precipitate forms on the bottom of a flask, add up to 10 mL of concentrated HCl to dissolve the precipitate. After precipitate is dissolved, dilute to volume with reagent water. Analyze by FLAA or ICP-AES.

7.6 Calculations

7.6.1 The concentrations determined are to be reported on the basis of the actual weight of the sample. If a dry weight analysis is desired, then the percent solids of the sample must also be provided.

7.6.2 If percent solids is desired, a separate determination of percent solids must be performed on a homogeneous aliquot of the sample.

8.0 QUALITY CONTROL

8.1 All quality control measures described in Chapter One should be followed.

8.2 For each batch of samples processed, a method blank should be carried throughout the entire sample preparation and analytical process according to the frequency described in Chapter One. These blanks will be useful in determining if samples are being contaminated. Refer to Chapter One for the proper protocol when analyzing method blanks.

8.3 Spiked duplicate samples should be processed on a routine basis and whenever a new sample matrix is being analyzed. Spiked duplicate samples will be used to determine precision and bias. The criteria of the determinative method will dictate frequency, but 5% (one per batch) is recommended or whenever a new sample matrix is being analyzed. Refer to Chapter One for the proper protocol when analyzing spiked replicates.

8.4 Limitations for the FLAA and ICP-AES optional digestion procedure. Analysts should be aware that the upper linear range for silver, barium, lead, and antimony may be exceeded with some samples. If there is a reasonable possibility that this range may be exceeded, or if a sample's analytical result exceeds this upper limit, a smaller sample size should be taken through the entire procedure and re-analyzed to determine if the linear range has been exceeded. The approximate linear upper ranges for a 2 gram sample size:

Ag	2,000 mg/kg
As	1,000,000 mg/kg
Ba	2,500 mg/kg
Be	1,000,000 mg/kg
Cd	1,000,000 mg/kg
Co	1,000,000 mg/kg
Cr	1,000,000 mg/kg
Cu	1,000,000 mg/kg
Mo	1,000,000 mg/kg
Ni	1,000,000 mg/kg
Pb	200,000 mg/kg
Sb	200,000 mg/kg
Se	1,000,000 mg/kg
Tl	1,000,000 mg/kg
V	1,000,000 mg/kg
Zn	1,000,000 mg/kg

NOTE: These ranges will vary with sample matrix, molecular form, and size.

9.0 METHOD PERFORMANCE

9.1 In a single laboratory, the recoveries of the three matrices presented in Table 2 were obtained using the digestion procedure outlined for samples prior to analysis by FLAA and ICP-AES. The spiked samples were analyzed in duplicate. Tables 3-5 represents results of analysis of NIST Standard Reference Materials that were obtained using both atmospheric pressure microwave digestion techniques and hot-plate digestion procedures.

10.0 REFERENCES

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TABLE 1

STANDARD RECOVERY (%) COMPARISON FOR METHODS 3050A AND 3050B[a]

Analyte	METHOD 3050A[a]	METHOD 3050B w/option[a]
Ag	9.5	98
As	86	102
Ba	97	103
Be	96	102
Cd	101	99
Co	99	105
Cr	98	94
Cu	87	94
Mo	97	96
Ni	98	92
Pb	97	95
Sb	87	88
Se	94	91
Tl	96	96
V	93	103
Zn	99	95

[a] All values are percent recovery. Samples: 4 mL of 100 mg/mL multistandard; n = 3.

TABLE 2

PERCENT RECOVERY COMPARISON FOR METHODS 3050A AND 3050B

	Percent Recovery[a,c]							
Analyte	Sample 4435		Sample 4766		Sample HJ		Average	
	3050A	3050B	3050A	3050B	3050A	3050B	3050A	3050B
Ag	9.8	103	15	89	56	93	27	95
As	70	102	80	95	83	102	77	100
Ba	85	94	78	95	b	b	81	94
Be	94	102	108	98	99	94	99	97
Cd	92	88	91	95	95	97	93	94
Co	90	94	87	95	89	93	89	94
Cr	90	95	89	94	72	101	83	97
Cu	81	88	85	87	70	106	77	94
Mo	79	92	83	98	87	103	83	98
Ni	88	93	93	100	87	101	92	98
Pb	82	92	80	91	77	91	81	91
Sb	28	84	23	77	46	76	32	79
Se	84	89	81	96	99	96	85	94
Tl	88	87	69	95	66	67	74	83
V	84	97	86	96	90	88	87	93
Zn	96	106	78	75	b	b	87	99

a - Samples: 4 mL of 100 mg/mL multi-standard in 2 g of sample. Each value is percent recovery and is the average of duplicate spikes.

b - Unable to accurately quantitate due to high background values.

c - Method 3050B using optional section.

Table 3
Results of Analysis of Nist Standard Reference Material 2704
"River Sediment" Using Method 3050B (µg/g ± SD)

Element	Atm. Pressure Microwave Assisted Method with Power Control	Atm. Pressure Microwave Assisted Method with Temperature Control (gas-bulb)	Atm. Pressure Microwave Assisted Method with Temperature Control (IR-sensor)	Hot-Plate	NIST Certified Values for Total Digestion (µg/g ±95% CI)
Cu	101 ± 7	89 ± 1	98 ± 1.4	100 ± 2	98.6 ± 5.0
Pb	160 ± 2	145 ± 6	145 ± 7	146 ± 1	161 ± 17
Zn	427 ± 2	411 ± 3	405 ± 14	427 ± 5	438 ± 12
Cd	NA	3.5 ± 0.66	3.7 ± 0.9	NA	3.45 ± 0.22
Cr	82 ± 3	79 ± 2	85 ± 4	89 ± 1	135 ± 5
Ni	42 ± 1	36 ± 1	38 ± 4	44 ± 2	44.1 ± 3.0

NA - Not Available

Table 4
Results of Analysis of NIST Standard Reference Material 2710
"Montana Soil (Highly Elevated Trace Element Concentrations)" Using Method 3050B
(µg/g ± SD)

Element	Atm. Pressure Microwave Assisted Method with Power Control	Atm. Pressure Microwave Assisted Method with Temperature Control (gas-bulb)	Atm. Pressure Microwave Assisted Method with Temperature Control (IR-sensor)	Hot-Plate	NIST Leachable Concentrations Using Method 3050	NIST Certified Values for Total Digestion (µg/g ±95% CI)
Cu	2640 ± 60	2790 ± 41	2480 ± 33	2910 ± 59	2700	2950 ± 130
Pb	5640 ± 117	5430 ± 72	5170 ± 34	5720 ± 280	5100	5532 ± 80
Zn	6410 ± 74	5810 ± 34	6130 ± 27	6230 ± 115	5900	6952 ± 91
Cd	NA	20.3 ± 1.4	20.2 ± 0.4	NA	20	21.8 ± 0.2
Cr	20 ± 1.6	19 ± 2	18 ± 2.4	23 ± 0.5	19	39*
Ni	7.8 ± 0.29	10 ± 1	9.1 ± 1.1	7 ± 0.44	10.1	14.3 ± 1.0

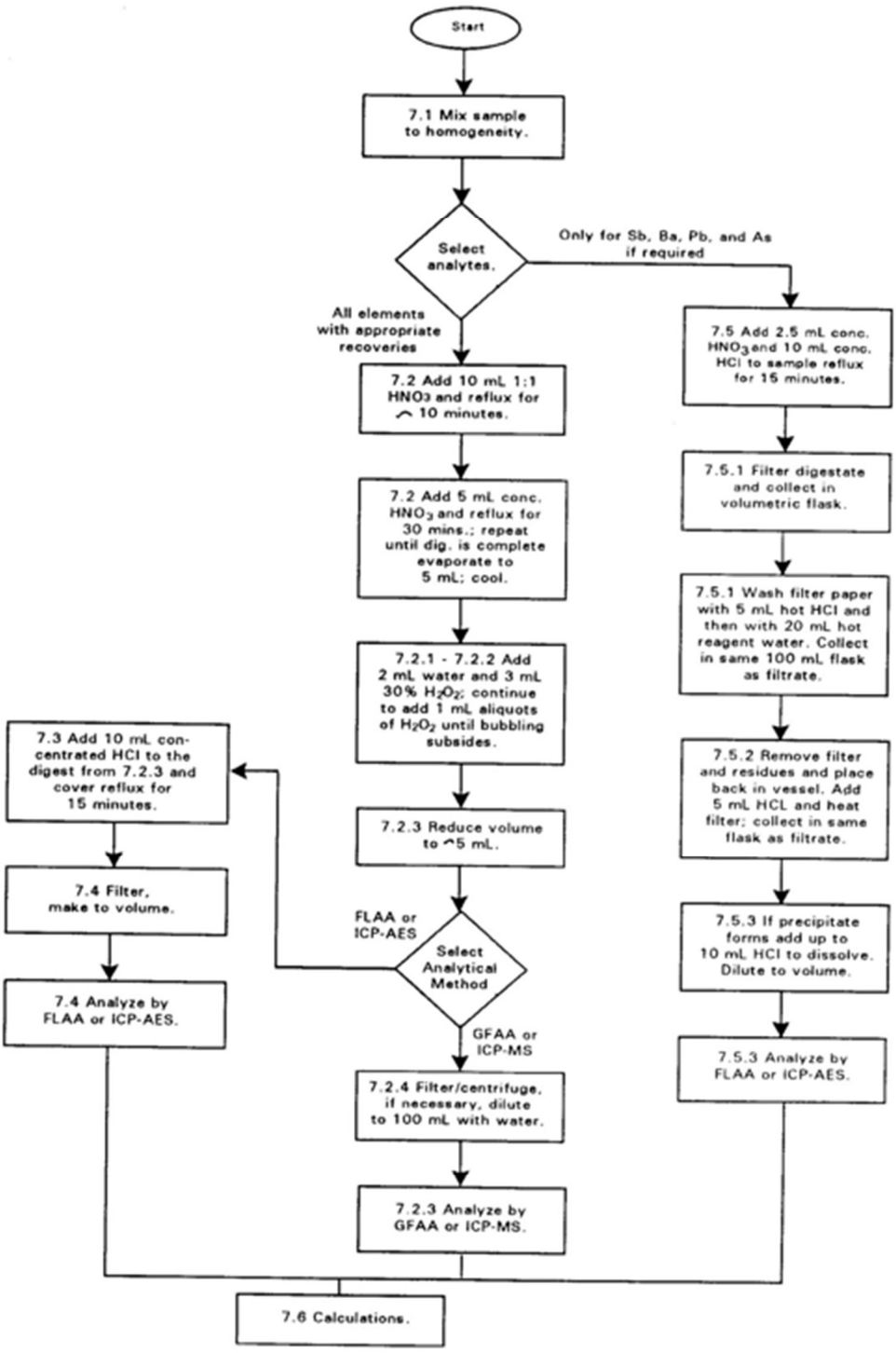
NA - Not Available * Non-certified values, for information only.

Table 5
Results of Analysis of NIST Standard Reference Material 2711
"Montana Soil (Moderately Elevated Trace Element Concentrations)" Using Method 3050B
(µg/g ± SD)

Element	Atm. Pressure Microwave Assisted Method with Power Control	Atm. Pressure Microwave Assisted Method with Temperature Control (gas-bulb)	Atm. Pressure Microwave Assisted Method with Temperature Control (IR-sensor)	Hot-Plate	NIST Leachable Concentrations Using Method 3050	NIST Certified Values for Total Digestion (µg/g ±95% CI)
Cu	107 ± 4.6	98 ± 5	98 ± 3.8	111 ± 6.4	100	114 ± 2
Pb	1240 ± 68	1130 ± 20	1120 ± 29	1240 ± 38	1100	1162 ± 31
Zn	330 ± 17	312 ± 2	307 ± 12	340 ± 13	310	350.4 ± 4.8
Cd	NA	39.6 ± 3.9	40.9 ± 1.9	NA	40	41.7 ± 0.25
Cr	22 ± 0.35	21 ± 1	15 ± 1.1	23 ± 0.9	20	47*
Ni	15 ± 0.2	17 ± 2	15 ± 1.6	16 ± 0.4	16	20.6 ± 1.1

NA - Not Available
* Non-certified values, for information only.

METHOD 3050B
ACID DIGESTION OF SEDIMENTS, SLUDGES, AND SOILS

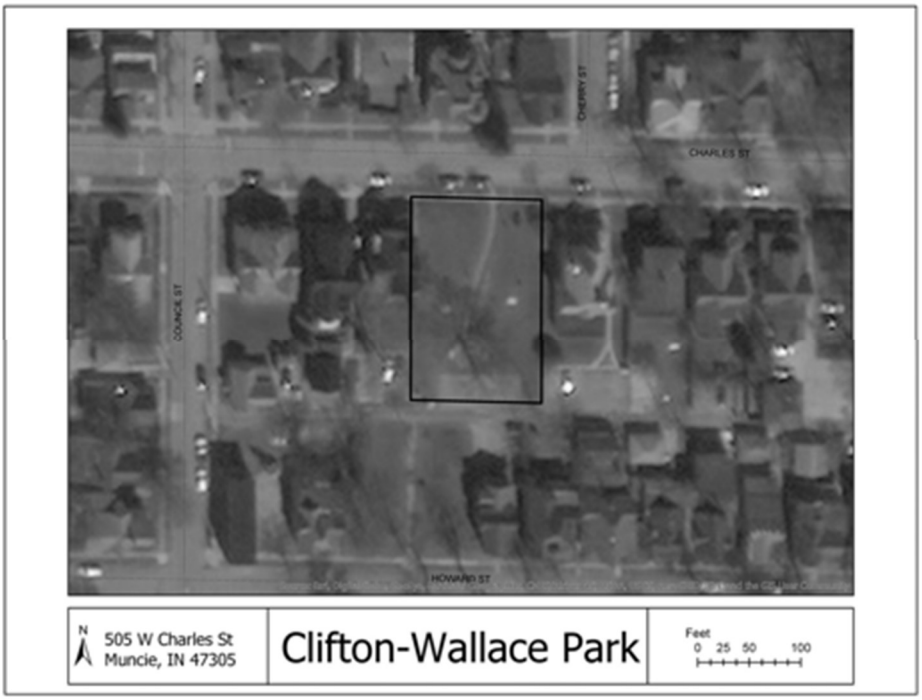


Appendix B

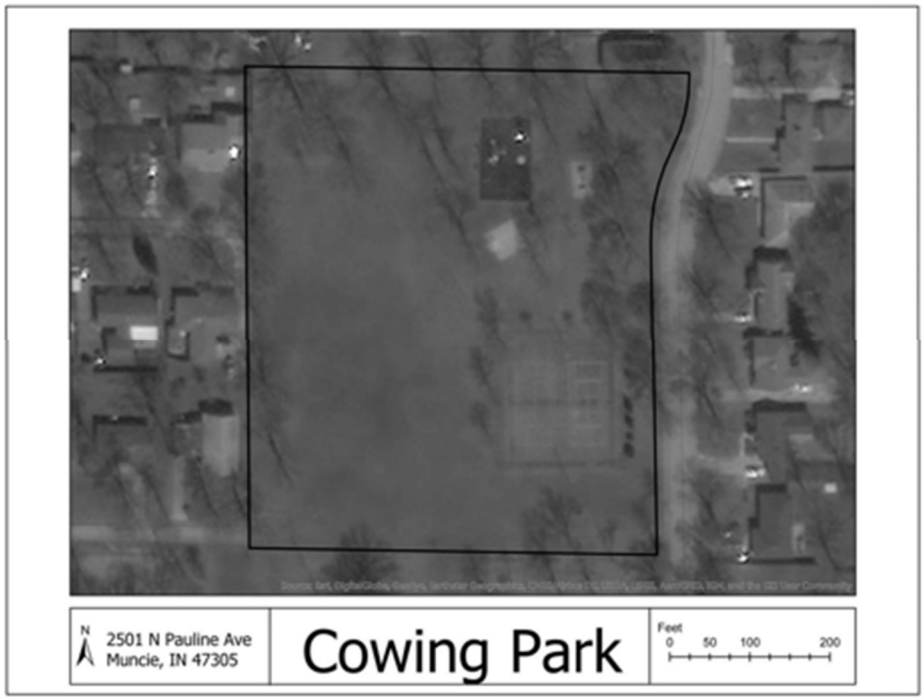
Site Maps

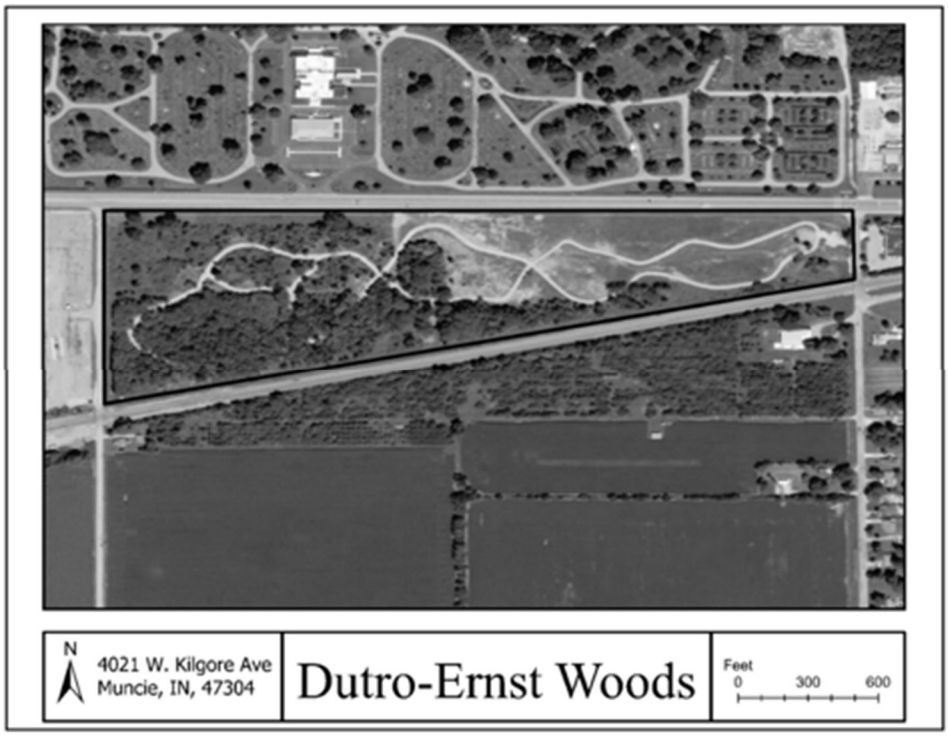

N
1900-1924 N Watt Ave
Muncie, Indiana 47303
Aultshire Park
Feet
0 50 100 200

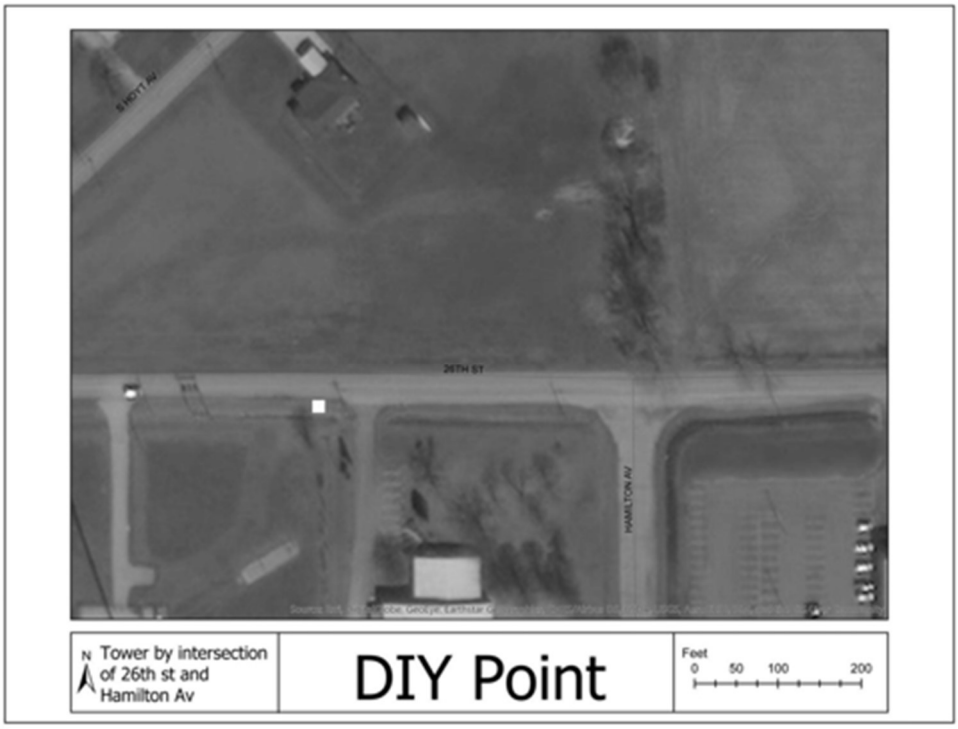

N
2201 S Macedonia Ave
Muncie, IN 47302
Ball Corp. Park
Feet
0 100 200 400


CHARLES ST
COUNCIL ST
HOWARD ST
N
505 W Charles St
Muncie, IN 47305
Clifton-Wallace Park
Feet
0 25 50 100


MOCK AVE
24TH ST
N
3120 S Mock Ave
Muncie, IN 47302
Cooley Park
Feet
0 50 100 200


N
2501 N Pauline Ave
Muncie, IN 47305
Cowing Park
Feet
0 50 100 200


N
4021 W. Kilgore Ave
Muncie, IN, 47304
Dutro-Ernst Woods
Feet
0 300 600

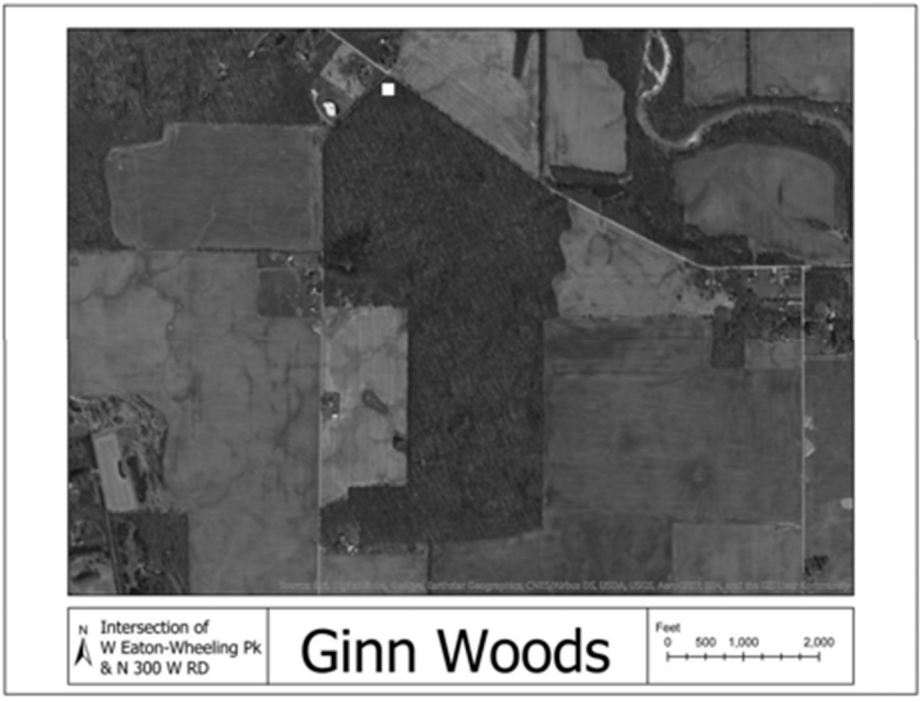

ELLIOTT AV
26TH ST
HAMILTON AV
N
Tower by intersection of 26th st and Hamilton Av
DIY Point
Feet
0 50 100 200


BEECHWOOD AVE
LINDEN ST
PAULINE AVE
ASHLAND AVE
N
900 W Ashland Ave
Muncie, IN 47303
Emerson Memorial Park
Emerson Dog Park
Feet
0 50 100 200


ELM ST
MADISON AVE
SR 32
N
Corner of Madison Av
& SR 32
Muncie, In
Fireman's Park
Feet
0 25 50 100


N
418 W Adams St.
Muncie, IN 47305
Friends Memorial Church
Feet
0 50 100 200


GILBERT ST
ELM ST
N
Corner of E Gilbert St.
North Elm Street
Muncie, IN 47303
Gilbert Park
Feet
0 20 40 80

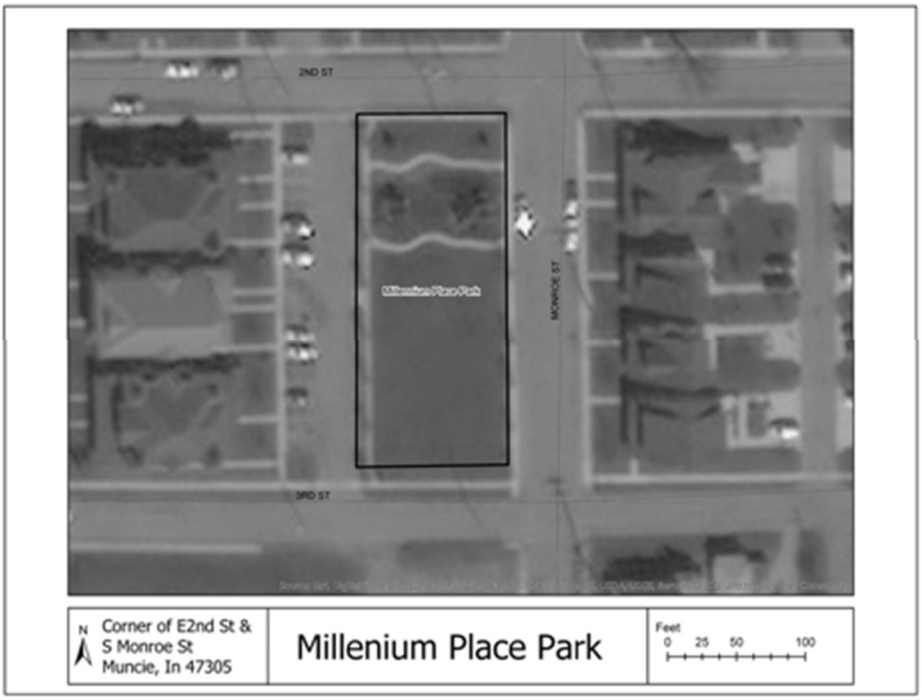

N
Intersection of
W Eaton-Wheeling Pk
& N 300 W RD
Ginn Woods
Feet
0 500 1,000 2,000


N
1800 S Grant St
Muncie, IN 47302
Heekin Park
Feet
0 125 250 500

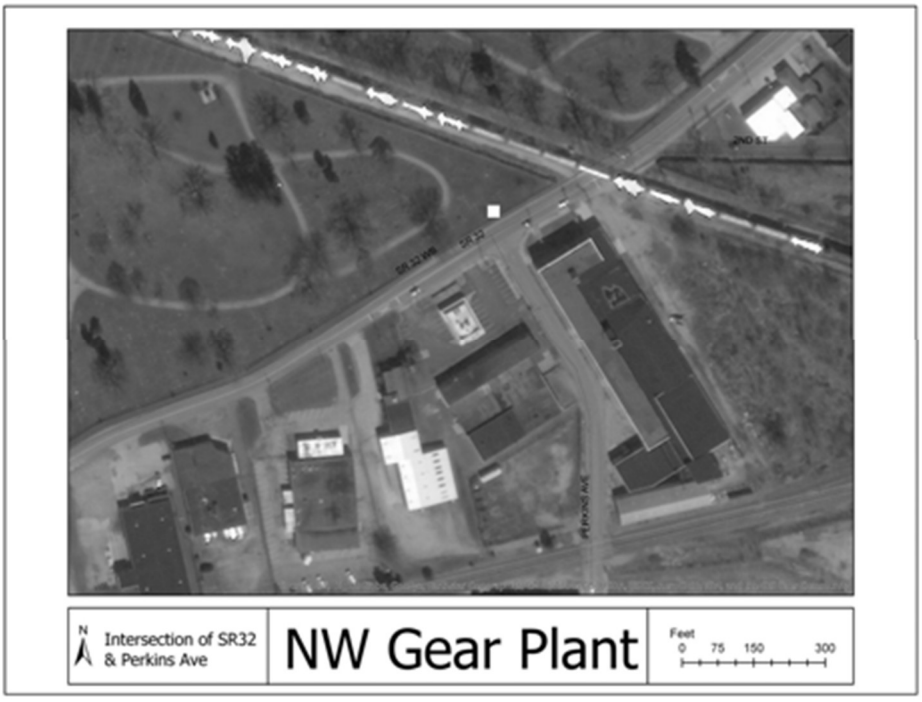

N
1400 W Uni Ave
Muncie, IN 47303
Mansion Point
Feet
0 75 150 300

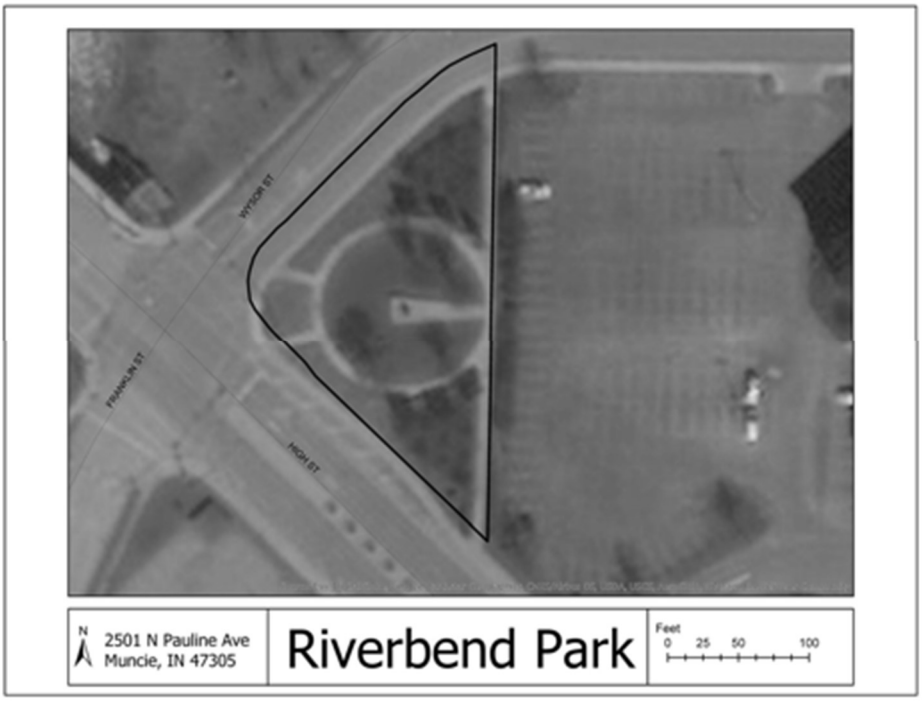

N
3800 N Glenwood Ave
Muncie, IN 47304
Matthews Park
Feet
0 50 100 200


N
1200 Dr. M.L.K. Jr Blvd
Muncie, IN 47303
McCulloch Park
Feet
0 375 750 1,500


2ND ST
MONROE ST
3RD ST
N
Corner of E2nd St &
S Monroe St
Muncie, In 47305
Millenium Place Park
Feet
0 25 50 100

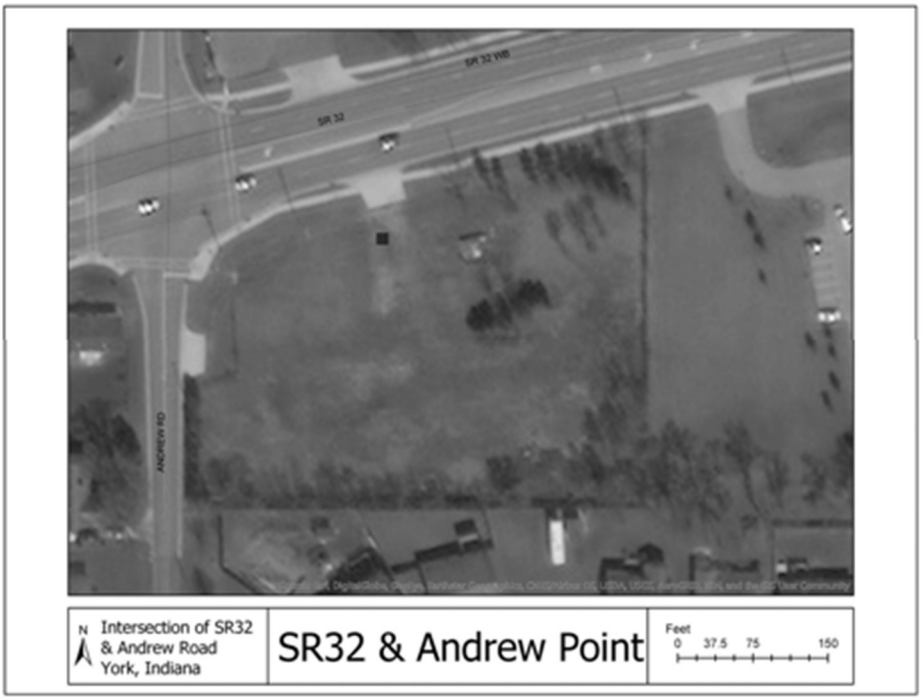

WALNUT ST
MULBERRY ST
NORTH ST
N
111 E North St
Muncie, IN 47305
Policeman's Memorial Park
Feet
0 25 50 100

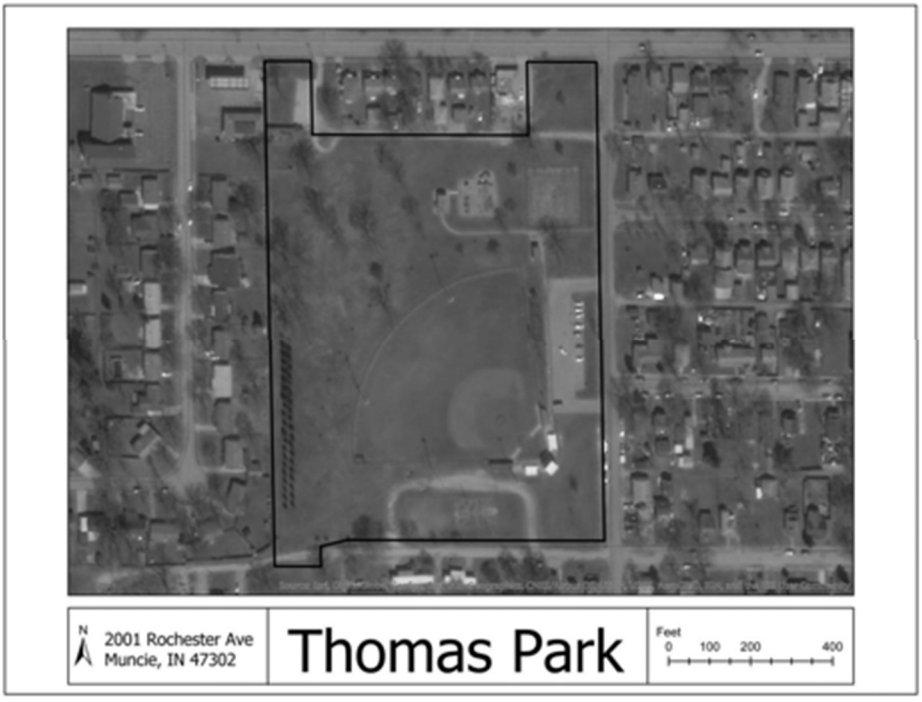

N
Intersection of SR32
& Perkins Ave
NW Gear Plant
Feet
0 75 150 300

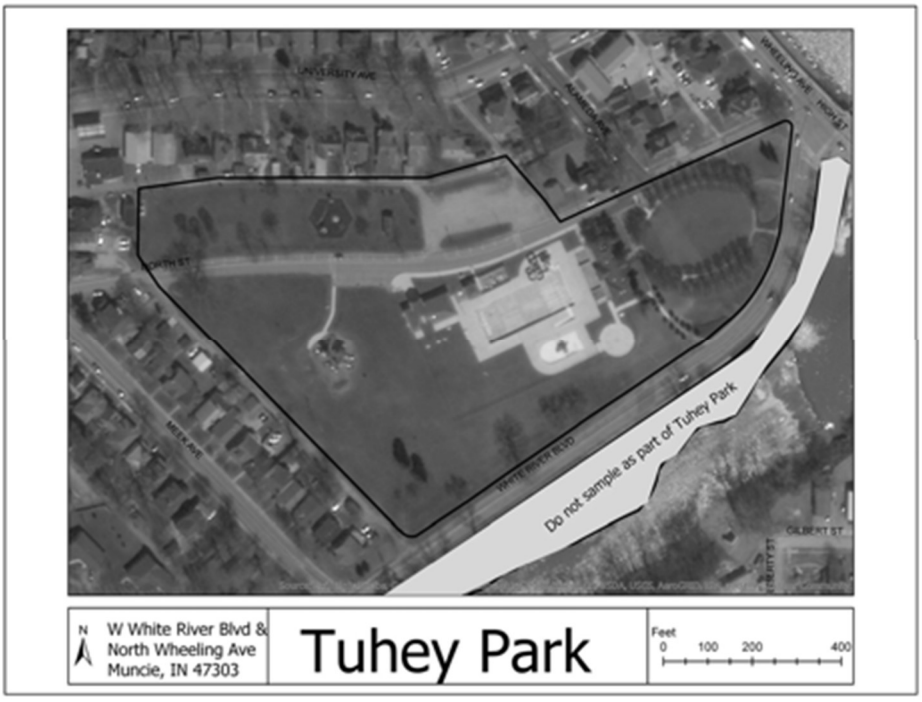

WYSOR ST
FRANKLIN ST
HIGH ST
N
2501 N Pauline Ave
Muncie, IN 47305
Riverbend Park
Feet
0 25 50 100


9TH ST
PIERCE ST
BIRCH ST
10TH ST
N
1110 W 10th St
Muncie, IN 47302
Rose Park
Feet
0 50 100 200


7TH ST
MOUND ST
ELLIOTT ST
EDYT AVE
8TH ST
BIRCH ST
ELLIOTT ST
9TH ST
N
Corner of S Elliott St
and W 7th St,
Muncie, IN 47303
SE GEAR PLANT
Feet
0 75 150 300


SR 32 WB
SR 32
ANDREW RD
N
Intersection of SR32
& Andrew Road
York, Indiana
SR32 & Andrew Point
Feet
0 37.5 75 150


N
2001 Rochester Ave
Muncie, IN 47302
Thomas Park
Feet
0 100 200 400


UNIVERSITY AVE
SUMMER AVE
WHEELING AVE
HOYT ST
26TH ST
MELN AVE
WHITE RIVER BLVD
Do not sample as part of Tuhey Park
GILBERT ST
N
W White River Blvd &
North Wheeling Ave
Muncie, IN 47303
Tuhey Park
Feet
0 100 200 400


ADAMS ST
VINE ST
PERSHING DR
CHARLES ST
N
304 S Pershing Dr
Muncie, IN 47305
Washington Park
Feet
0 30 60 120

Bureau, U. S. C., 2018, TIGER/Line Shapefile, *in* Bureau, U. S. C., ed.
DTSC, 2011, Lead Risk Assessment Spreadsheet 8, *in* Control, C. D. o. T. S., ed.
HERO, 2019, Human Health Risk Assessment Note 3, *in* Control, C. D. O. T. S., and Office, H. A. E. R., eds., p. 1-36.
IGAW, 2020, IndianaMAP.